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Westinghouse Savannah River Company

Interim Report for Crucible-Scale Active Vitrification of Waste Envelope B (AZ-102) (U)

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Savannah River Site
Aiken, SC 29808



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**Interim Report for Crucible Scale Active Vitrification of Waste
Envelope B (AZ-102) (U)**

SAVANNAH RIVER TECHNOLOGY CENTER

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Interim Report for Crucible-Scale
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SUMMARY

As part of the Hanford River Protection Project-Waste Treatment Plant (RPP-WTP), the Savannah River Technology Center (SRTC) has produced an Immobilized Low Activity Waste (ILAW) glass from a pretreated, decontaminated Hanford Tank 241-AZ-102 sample. The glass was made from a radioactive supernate that was pretreated by SRTC personnel using ion exchange to remove Cs-137 and Tc-99. The AZ-102 supernate was originally prepared from filtration of a raw ~ 3.75 L tank sample (mostly supernate with small amounts of sludge solids) provided by Hanford to SRTC. The AZ-102 ILAW glass was produced by first concentrating the decontaminated supernate in a vacuum evaporation apparatus at a target temperature of 50°C. The actual vacuum and temperature observed near the evaporation endpoint was 60 mm Hg (-27.4 inches Hg) and 49.7 °C. The evaporator feed supernate (~ 600 mL) was concentrated by a factor of 1.9 based on soluble analyte concentrations measured in the starting evaporator feed and ending concentrate (~ 305 mL). This concentration factor of ~1.9X was based on OLI model calculations to reach 80% bulk saturation. No solids formation in the concentrated evaporator concentrate was observed after completion of the evaporation.

The AZ-102 evaporator feed material was analyzed and the characterization results were used by Vitreous State Laboratory of Catholic University of America (VSL of CUA) to provide a glass formulation recipe targeted at 5 wt% Na₂O in glass. The expected sulfur content in the glass was 0.8 wt% measured as SO₃. The concentrated supernate waste stream was mixed with a blend of 10 glass forming minerals in a platinum/gold crucible and heated to 1150 °C. The glass melts were then cooled following a prescribed cooling curve. Visual inspection of the two resulting replicate ~ 110 gram glasses indicated secondary phase formation on the bottom of each glass. This phase was analyzed to be an unnamed clinopyroxene, a multiple cation (Ca²⁺, Na⁺, Mg²⁺, Fe³⁺, Cr³⁺, Al³⁺) silicate. Another replicate radioactive AZ-102 glass was vitrified and allowed to 'quench cool'. This glass did not contain any secondary phase. The glass was analyzed to contain near target specified amounts of glass components and no crystalline phases were observable with x-ray Diffraction (XRD) analysis. Sulfur analyses on the glass was not included in these preliminary glass analyses. The remaining portion of this quench-cooled glass was remelted and centerline cooled to determine if a secondary phase developed on slow cooling. The secondary clinopyroxene phase (augite), a multiple cation (Ca²⁺, Na⁺, Mg²⁺, Fe^{2+/3+}, Al³⁺) silicate enriched in Fe²⁺ was determined on the bottom of this glass, although to a lesser extent than was previously observed in the first two replicate tests.

Studies with nonradioactive oxidized surrogates supplied to SRTC by VSL have also produced glasses with XRD-detectable augite phases on the bottom portion of the glass. This was observed in both dry powder melts and remelts of glass produced at VSL. These surrogate glasses were also slow cooled by centerline cooling schedule. The crystalline phase on these glasses made with VSL simulants were significantly less visible than the initial radioactive feed crucible melts.

A revised simulant of the AZ-102 concentrate was prepared based on re-characterization data obtained on the radioactive AZ-102 feed at SRTC. This simulant was formulated to contain several grams per liter amounts of various dissolved organic carbon components such as acetate, citrate, formate, glycolate and EDTA. Crucible scale vitrification tests with this simulant have

replicated the quantitative and qualitative crystalline secondary phases observed with the initial crucible melts of radioactive feed, e.g., the Fe²⁺ enriched augite. Preliminary testing with this simulant in different crucible melt configurations suggests that increased air flux in the crucible melts influences the amount of visible crystalline phase present on the bottom of the glass melts.

The following conclusions can be reached from crucible vitrification testing performed at SRTC to date:

- The secondary crystalline phase observed on the bottom of radioactive glass melts is reproducible and only forms on canister cooling of the glasses. In the case of the majority of crystalline phases identified, it is a Fe²⁺ enriched species (augite) likely stabilized by the organics in the waste.
- No evidence of a secondary phase was observed when the radioactive melt was rapidly cooled. However, a secondary phase was observed when this rapid quenched glass was remelted and allowed to canister cool.
- Two examples are cited which show the redox state of the glass could be related to the presence of the secondary crystalline phase. One example involves comparing the VSL oxidized simulant glasses with low observable crystalline secondary phase and no added organic carbon to the SRTC simulant glasses with high amounts of crystalline secondary phase and total organic carbon present at ~ 10 g/L. The second example of the potential effect of redox on the glasses is the observed decrease in visible secondary crystalline phase, i.e., less crystals on the bottom of the glass surfaces, with increasing air flux during the melt and cooling cycles.
- Scanning Electron Microscopy data shows presence of platinum associated with the secondary crystalline phase. The platinum could be linked to the formation of the secondary crystalline phase, e.g., as a nucleating agent, but not to the amount of the phases as a function of redox.
- No evidence of any sulfur-containing phase has been observed from the x-ray diffraction and Scanning Electron Microscopy data. (Note: No sulfur measurements have been made to date on the glasses since sulfur analyses in the glasses were planned as part of the Regulatory Analyses that have not been undertaken at this time due to the observed crystallinity in the radioactive glasses made to date).

Observation of the secondary phase clinopyroxene on the canister-cooled glasses (and not on the rapid-cooled, quenched glasses) suggests that this secondary phase should have no significant effects on the operation of the LAW melter. This is because the glass in the melter is at molten melt temperature of ~ 1150 °C and is poured out of the melter before the secondary crystalline phase forms upon controlled cooling. Observation of the secondary phase clinopyroxene on the canister-cooled glasses could possibly have significant effects on the glass durability since it is well accepted that certain crystalline glass phases can leach at a higher rate than amorphous, non-crystalline glass.

Given the above data and conclusions, the current path forward for this Envelope B, AZ-102 sample is to revise the glass formulation with surrogate testing at VSL/CUA, followed by further crucible-scale vitrification testing with the remaining decontaminated AZ-102 radioactive sample at SRTC. The goal would be to produce an amorphous AZ-102 glass with no significant crystal structure present. The revised formulation product glass made from the decontaminated radioactive sample will be produced and tested per a new Task Specification and Task Plan that are currently in development by Bechtel/Washington Group and SRTC personnel.

INTRODUCTION

The Department of Energy, Office of River Protection is utilizing subcontractors to design, construct, and operate facilities to immobilize radioactive waste stored in underground tanks at the Hanford site near Richland, Washington [1,2]. The program is called the River Protection Project-Waste Treatment Plant (RPP-WTP). Present studies are a continuation of previous collaborative efforts of BNFL, Inc. and the Savannah River Technology Center (SRTC) [3]. During Part B-1 SRTC research and testing demonstration, the Immobilization Technology Section (ITS) of SRTC has demonstrated, using a crucible-scale furnace, the vitrification portion of the process to producing an ILAW glass waste form from Hanford Tank 241-AZ-102. This small active vitrification task evaporates and vitrifies samples from radioactive waste treatment demonstrations that were performed by SRTC as part of a Work for Others (WFO) agreement [1,4].

The purposes of this work were to demonstrate the evaporation of AZ-102 supernate, demonstrate the vitrification of the evaporated concentrate in a crucible melt, and to demonstrate acceptance of the resulting glass by analysis (chemical and radionuclides) and durability testing. This is an interim report for the small-scale active crucible vitrification testing on the AZ-102 sample. RPP-WTP personnel have outlined the information that they are expecting from this study in a previous Task Specification document [4]. SRTC issued the task planning document to address the task specification document [5]. The scope of this report includes results from preliminary testing with the pretreated AZ-102 radioactive feed. Details on the characterization, filtration and ion exchange of the original AZ-102 raw tank sample are described elsewhere [6,7]. The AZ-102 characterization data was supplied to the VSL for glass formulation development. The resulting formulation was used to produce several radioactive crucible melts at SRTC. Further crucible vitrification testing was pursued with different surrogate feeds upon observation of a secondary crystalline phase formation in the radioactive AZ-102 glass products. Data from production of several radioactive AZ-102 glasses and surrogate glasses are presented. This report does not include the original scope of glass analyses and glass durability testing due to the change in scope upon observation of the crystalline material in the glasses.

EXPERIMENTAL

Feed Stream Evaporation and Waste Glass Formulation

The goal of the Feed Stream Evaporation and Waste Glass Formulation phase was to preconcentrate the decontaminated Envelope B liquid by a factor of 1.9X and mix the waste form and glass-forming chemicals in 600-mL platinum/gold crucibles. The radioactive AZ-102 crucible melts were planned to be analyzed by regulatory analyses to include metals, radionuclides, organics, halides and sulfur, and performance testing (Product Consistency Testing and Toxicity Characteristic Leaching Procedure) [8]. Initial Contract specifications did not include vapor hydration testing for the LAW glass

products. The expected concentration endpoint of 80% of bulk saturation @ 25°C was determined based upon the simulant evaporation studies and OLI modeling discussed in Reference [9]. The pretreated AZ-102 LAW supernate feed was evaporated to the predicted concentration endpoint. The concentrated pretreated LAW was mixed with glass-forming chemicals to complete the feed stream preparation phase. Except for preparation of supplies and glass-forming chemicals, the feed preparation was performed in radiochemical hoods within a radiological buffer area (RBA).

Decontaminated Liquid Feed Evaporation

A schematic of the evaporator is shown in Figure 1. This 'pot' evaporation unit was operated in a single batch mode, *i.e.*, no continuous feed. This apparatus is designed to incorporate some of the same design elements used in a concurrent bench scale LAW simulant evaporation program ongoing at SRTC [9]. Evaporations were performed at reduced pressure (40 – 80 Torr absolute pressure) provided by a diaphragm pump at nominally 50 °C. Preliminary leak testing in the setup showed approximately 1.5 inches of Hg pressure loss over a 24-hour period. This air inleakage equals 1.6E-03 atm cm³/s, or about 0.21% of system volume. Both the primary and secondary chillers in the evaporation unit were cooled by separate water chiller units.

Liquid evaporation equipment included a central-placed heater rod (Watlow FIREROD, 3/8" diameter x 18" length, equipped with Type K thermocouple), vacuum pump (Vacuubrand chemistry diaphragm pump, Model MZ 2C), voltage and current monitoring device (Cole-Parmer DC/AC Model 26840-20 Multimeter), pressure gauge (Ashcroft ASME Test Grade 2A, 0.5% accuracy) and temperature measuring devices (Cole-Parmer Digi-Sense platinum Resistance Temperature Device (RTD) thermometry systems). The pressure gauge, heater rod thermocouple, voltage and current monitoring device, and RTD thermometry systems were all calibrated before use by the SRTC standards lab to National Institute of Standards and Technology (NIST)-traceable standards. Results of these calibrations are routinely maintained by a calibration coordinator in the Immobilization Technology Section (ITS) of SRTC.

Condensate was collected below the primary condenser and the concentrate was collected in the large heated pot. After the batch evaporation experiment, the system was allowed to cool to ambient temperature. Both concentrate and condensate samples were then obtained and analyzed according to Table 1 shown below.

Power to the heated rod was controlled via a variable power supply. Both voltage and current input to the heating rod was monitored and controlled. Evaporation details such as endpoint targets and operating procedures were determined from other ITS studies involving evaporation experiments on simulant streams [9]. Control parameters that were monitored during evaporation activities were pot temperature, pressure, voltage, current and condenser exit temperatures.

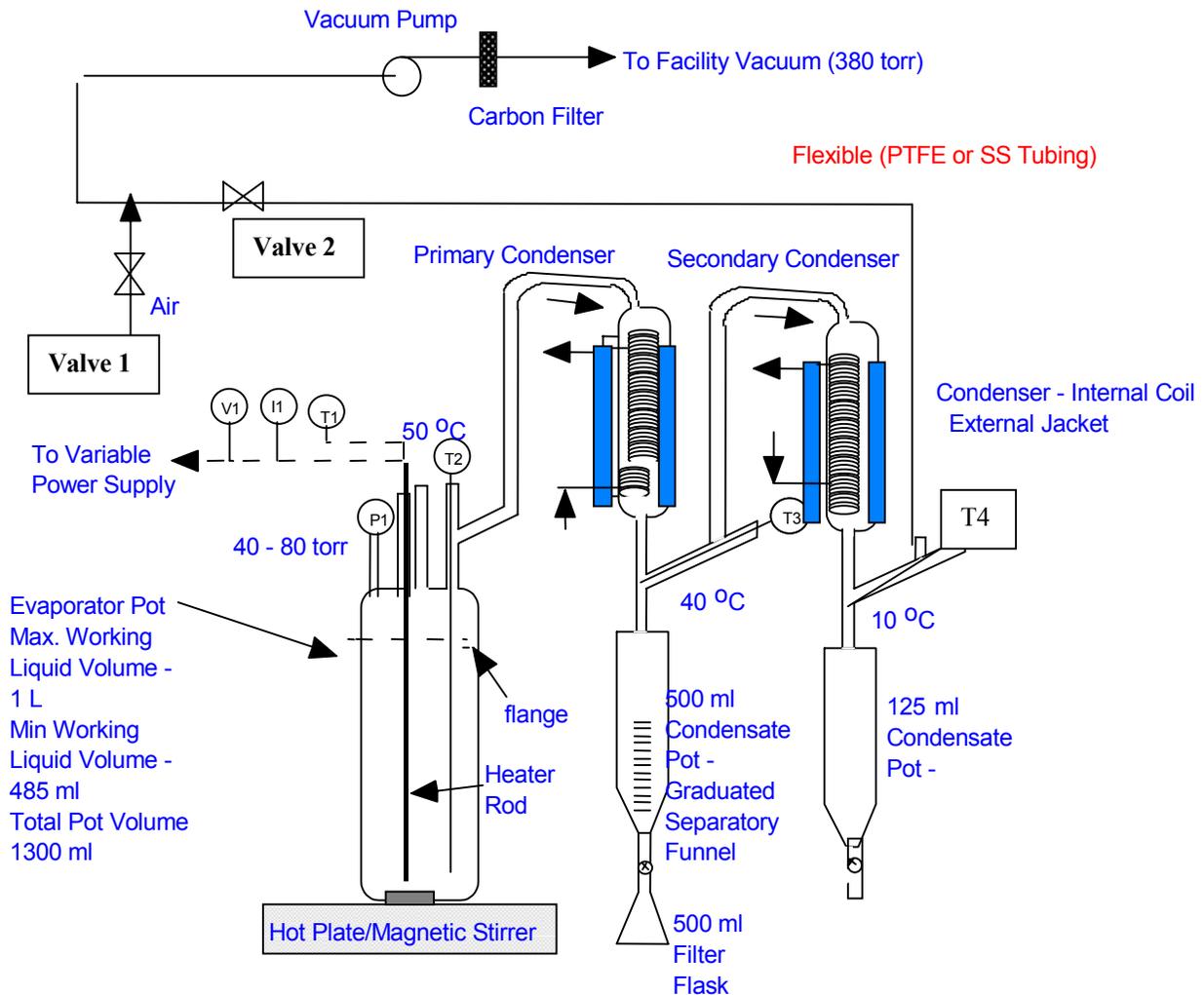


Figure 1. Small LAW Pot Evaporator

Notes:

- V1/I1 = Variable voltage and current input to heating rod
- P1 = Vacuum pressure gauge
- T1 = Type K thermocouple temperature measurement
- T2 = RTD temperature readout in Concentrate Pot
- T3, T4 = RTD temperature measuring devices in primary and secondary condenser exits, respectively
- Valve 1 = air inleakage port controller
- Valve 2 = system vacuum on/off

Table 1. Required Analytical Support

Technique	Characterize Concentrate and Condensate	Sample Preparation (Glass Formers)	PCT Leach** Tests	Glass Analyses**
Na₂O₂ Fusion		X		X
HNO₃ Dissolve				X
ICP-ES	X	X	X	X
AA(Na/K)	X			X
ICP-MS	X			X
IonChromatography -Anions	X			
Chemchek = Total Uranium				X
Gamma-PHA	X			X
Alpha-PHA				X
Beta-Scint.	X			X
Liquid Scint.				X
Sr-90	X			X
Tc-99	X			X
TIC/TOC	X			
XRD				X
SEM/EDAX				X
Weight% solids	X			
AlO₂ + CO₃ + Free OH	X			

* These analyses are to be performed at SRTC. Separate regulatory analyses are planned [8].

** Only limited glass analyses have been performed to date on the AZ-102 crucible melts due to the secondary crystalline phase observations (see text).

ICP-ES = Inductively Coupled Plasma Emission Spectroscopy

AA(Na/K) = Atomic Absorption Spectroscopy for Na and K

ICP-MS = Inductively Coupled Plasma Mass Spectroscopy

Gamma-PHA = gamma-ray spectroscopy pulse height analysis

Alpha-PHA = alpha particle spectroscopy pulse height analysis

Beta-Scint. = beta scintillation counting

Liquid Scint. = liquid scintillation counting

TIC/TOC = total inorganic carbon / total organic carbon

XRD = x-ray diffraction

SEM/EDAX = Scanning Electron Microscopy/Energy Dispersive X-ray Analysis

Waste Glass Formulation

Appropriate amounts of decontaminated and concentrated AZ-102 liquid waste and a nonradioactive glass-forming chemical stream were mixed directly in the Pt/Au crucibles. All glass-forming chemicals were comprised of various mineral compositions specified by VSL. Previous studies in Part A work used reagent grade chemicals as the glass-forming chemicals [3]. Ongoing developmental work performed with waste simulants at VSL at CUA and at SRTC [10] supports this task. The results from the VSL development work form the basis for the formulations used in this step. The types and forms of industrial grade glass formers used in this study are the same as those used in the VSL development to facilitate comparison of the glasses made from simulated and actual waste. Appendix A contains detailed information on each glass former mineral used.

Concentrated AZ-102 supernate was analyzed in duplicate according to the analyses shown in Table 1. Results from these analyses were transmitted to VSL, who then specified the appropriate amounts of the waste streams, composition of the glass-forming chemicals, and appropriate amounts of the glass-forming chemicals to SRTC. RPP-WTP personnel reviewed the recommendations and approved them before SRTC blended the glass formers. As an additional quality assurance step, SRTC analyzed a representative portion of the final glass formers according to Table 1, and confirmed that analyzed components for Al, B, Ca, Li, Fe, Mg, Si, Ti, Zn, Zr agreed with as-batched values before actual mixing of the glass forming chemicals and decontaminated feed stream. Limited analyses for certain RCRA metals (Ba, Cd, Cr, Pb, V) are also presented.

The final mixtures of glass-forming chemicals and pretreated concentrated AZ-102 feed were prepared in 600-mL platinum/gold crucibles for the batch vitrification tests. The specified amount of blended glass formers were weighed and transferred to the Pt/Au crucible. The target amount of AZ-102 feed was measured by volume using graduated cylinders and delivered to the Pt/Au crucibles. Limited stirring with a spatula was used to mix the dry powder glass formers with the liquid AZ-102 feed. Balances used to measure the glass-forming chemicals were standard, single-pan, top-loading analytical balances calibrated by the SRTC standards lab using NIST-traceable standards. Calibration checks were performed with standard masses that bracketed the mass of the material to be weighed prior to all weighings.

Vitrification

The goal of the Vitrification phase was to immobilize the AZ-102 waste stream in a glass matrix that could then be characterized and performance tested. The target melt temperature for all active crucible scale vitrifications was 1150°C. The AZ-102 waste stream is a Resource Conservation and Recovery Act (RCRA) listed waste. Therefore, a special offgas treatment system was used for the vitrification testing to treat and remove some of the hazardous constituents so that the resulting offgas effluent could be released to the SRTC exhaust system. Offgas condensates were collected for residual return to

Hanford. Evaporation and calcining occurred in the furnace at temperatures from 100°C to 900°C. Heat-up rates during these steps were nominally < 100°C/hour. The furnace temperature was then increased from 900°C to 1150°C at a nominal heat-up rate of 100°C/hour. The vitrification temperature of 1150°C was held for 4 hours. After vitrification, the glass waste form was cooled according to a prescribed cooling schedule provided by VSL/RPP-WTP.

Waste Form Evaporation and Calcining

After mixing the glass-forming chemicals with the pretreated, concentrated waste in the vitrification crucible and transferring the resulting slurry to the vitrification furnace, water from the slurry was slowly removed by evaporation. This process is described in more detail below. A detailed diagram of the furnace as configured for these studies is presented in Figure 2. The furnace used for these studies is a DelTech Model DT-29-TL-610 Top Loading Laboratory Furnace capable of 1200°C with a programmable setpoint temperature control. The furnace was initially 'baked out' and calibration-tested before use according to recommended procedures by the vendor. Thermocouples and digital readouts used for calibration of the furnace were calibrated by the SRTC standards lab with NIST-traceable standards.

An offgas collection glassware apparatus was attached to the quartz glass system within the furnace. This offgas system contained and trapped all gaseous hazardous species evolved during the slurry evaporation, calcine and vitrification steps. The offgas system contains a primary water-cooled condenser, a dry ice bath and two activated carbon beds in series. A collection pot was connected to the water condenser for final collection of all condensed phase liquid collected during the evaporation and calcine steps. As shown in Figure 2, ambient air flowed into a quartz tube through an inlet carbon filter. The quartz vessel inside of the furnace contained an alumina insert that held the vitrification crucible. Incoming air will swept through the quartz tube carrying offgas from inside the sealed quartz vessel system to the offgas system (condenser, cold trap and carbon filters). The central offgas tube exits the furnace through a 1" diameter opening cut out of the top of the furnace. All loading of equipment and samples into the furnace was performed through a top-located circular furnace door of 6" diameter (not shown in Figure 2).

The final carbon filter in the offgas system was connected to vacuum. A vacuum of nominally 2-3 inches of water was maintained on the crucible throughout the entire vitrification process. The vacuum was monitored periodically by connecting a water manometer to the air inlet. Vacuum was supplied by SRTC facility-supplied vacuum through a connection within the radiochemical hood. Temperatures of the furnace were slowly increased to remove the water from the slurry contained in the crucible and collect it in the trap below the condenser. The nominal heatup rate was 10°C/hour. After the water was evolved from the waste form, the volatile species were driven off by slow temperature increases typically between 200 °C and 900 °C. Initial planning required use of a 250-mL crucible. However, due to increased amounts of glass product needed for all planned analyses [8], a 600-mL platinum/gold crucible was used.

(Offgas exits to: (1) water condenser, (2) dry ice bath, (3) carbon filters in series)

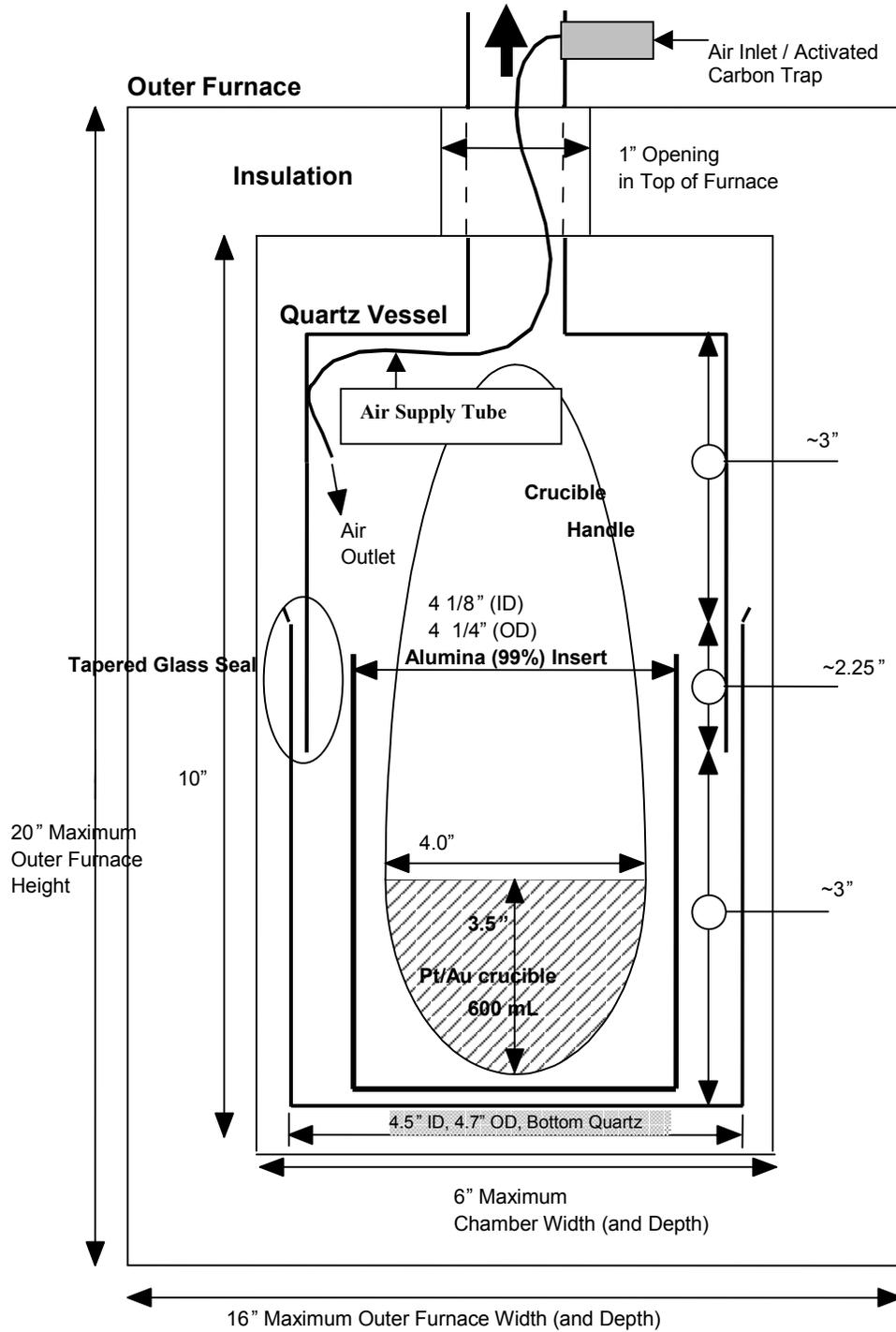
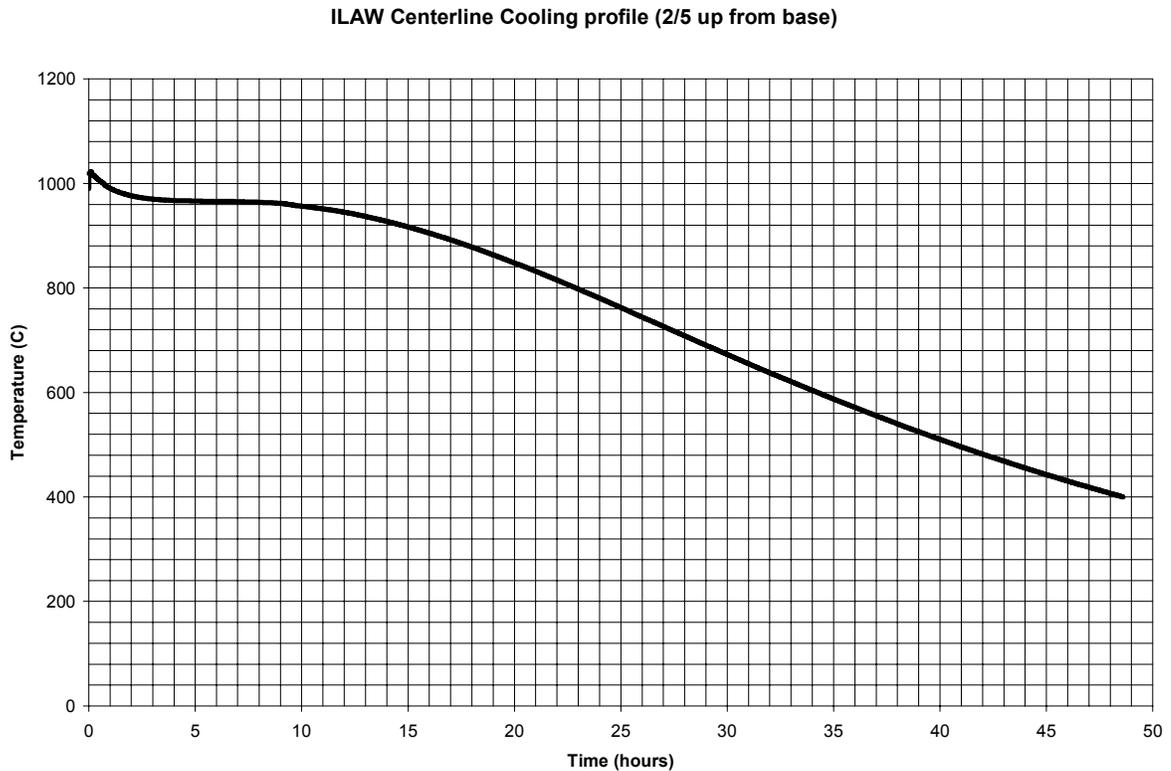


Figure 2. Small Active Crucible-Scale Furnace

Waste Form Vitrification, Cooling and Removal

The waste form was vitrified in the same furnace used to calcine the waste form. The temperature was slowly increased from 900°C, to the melt temperature of 1150°C, where it was held at this temperature for ~ 4 hours. After the melting period, the molten glass was cooled inside of the furnace according to the cooling schedule provided by VSL/RPP-WTP [LAW Cooling Curve Source: E-mail sent to B. Calloway (SRTC) on 11/15/99 from S. Arm (formerly with BNFL, Inc.) CH2MHill Hanford Group Reference # is 009325]. The glass cooling profile was intended to simulate the temperature cooling profile of glass in containers planned for the RPP-WTP. The temperature range for the controlled cooling was 1100 °C to 400 °C. This cooling profile is shown in Figure 3. After the furnace program reached the lower temperature of the cooling schedule, heating of the furnace was discontinued and the system was allowed to cool to ambient temperature. The glass and offgas system were handled after the furnace vitrification system was at ambient temperature. Liquids from the condensate traps were collected and their amounts measured by volume. No analyses were planned for any of the small active vitrification condensates resulting from crucible-scale furnace vitrification activities.

Figure 3. Cooling Curve for AZ-102 Vitrification



Glass Dissolution and Analyses

The initial two radioactive glasses produced from AZ-102 (Tests #1 and #2) were not analyzed due to the observed secondary phase on the bottom of the glasses. A third replicate radioactive glass was made (Test #3) with 'quench-cooling'. (Note: See Results and Discussion Section below for details of each crucible vitrification Test). This Test #3 glass, which did not have a visible secondary phase, was pulverized to a (-) 200 mesh size using a Mixer Mill with agate cups and agate grinding ball. Resulting glass powders were verified to be (-) 200 mesh by passing through an ASTM-certified brass sieve. These powdered glass samples were then dissolved using versions of ASTM glass dissolution procedures involving $\text{Na}_2\text{O}_2/\text{NaOH}$ fusion with acid uptake (ASTM C 1317-95), and acid dissolution (ASTM C 1412-99).

The peroxide fusion method used nominally 0.5 gram powdered glass samples added to 3 grams of Na_2O_2 and 2 grams of NaOH in new Ni-crucibles. The resulting mixture was heated in a Thermolyne furnace at 700 °C for 15 minutes. The resulting mixture was then cooled and transferred to a 100-mL volumetric plastic flask. A volume of 25 mL of concentrated 15.7 Molar nitric acid was used to rinse the crucible and also added to the flask. The sample was then diluted to the 100-ml mark of the volumetric flask.

The acid dissolution method used nominally 0.5 gram powdered glass sample added to an I-CHEMTM certified wide mouth plastic bottle. Then 10 mL of 50% (~ 29 Molar) HF and 10 mL of concentrated 15.7 Molar HNO_3 and 70 mL of 0.6 Molar boric acid solution were added. The bottle was capped and the mixture was heated in an oven at 105 °C for 2 hours. The mixture was then cooled, 70 mL of 0.6 Molar boric acid was added to the wide mouth plastic bottle, re-sealed and heated for an additional hour. After cooling, the solution was diluted to 100 ml in a volumetric flask with deionized water.

As previously mentioned in the notes to Table 1, the dissolved Test #3 quench-cooled glass was only analyzed for major chemical components using ICP-ES to determine if the product glass agreed reasonably well with the target formulation.

All other crucible glasses produced in this study were not subjected to dissolution and chemical characterization. Glasses produced in Tests #2 - #9 were analyzed by XRD and SEM/EDAX. The XRD analyses were performed on either pulverized glass powder or the top and bottom surfaces of the glass pucks. SEM/EDAX was performed on crushed glass powder for Test #3, on small shards of the glass from Test #2 and on the top and bottom surfaces of glasses from Tests #4 - #9. SEM analyses typically use two different methods of backscatter and secondary electrons. The backscatter image involves incident electrons that indicate potential density differences in the image particles. Use of secondary electron imaging that involves actual electrons from the glass matrix material provides topography images of the matrix.

RESULTS AND DISCUSSION

Feed Stream Evaporation and Preparation

Decontaminated Liquid Feed Evaporation – Active AZ-102 Sample

The pretreated AZ-102 supernate was prepared in early calendar year 2000. A total volume of ~ 600 mL resulted after initial characterization [6] and ion exchange pretreatment for Cs-137 and Tc-99 removal [7]. Table 2 shows the analytical characterization of the AZ-102 evaporator feed [7]. Cesium-137 was analyzed by gamma spectroscopy pulse-height analysis and other radionuclides were detected (Tc-99 and some actinides by ICP-MS and Pu-isotopes by separation and counting). It should be noted that the other gamma-emitting radionuclides shown in Table 2 (Co-60, Eu-154, Eu-155 and Sb-125) are reported at less than the instrument detection (minimum detectable activity (mda)) limit. These mda values are the upper limit estimate of these radionuclides that could be detected in the presence of quantifiable Cs-137. The average values from Table 2 were transmitted to Alex Choi of the Immobilization Technology Section for mathematical modeling to predict the endpoint and operating parameters for the evaporation of AZ-102 evaporator feed using “ESP” software developed by OLI Systems Inc., Morris Plains, NJ. Table 3 below summarizes the calculation results to determine water removal based on OLI calculations. The model predicted that to reach 80% of saturation 48.0% of water from the radioactive pretreated AZ-102 feed would have to be removed, concentrating it to 21.5 wt% total solids. The steady-state operating pressure for the 50 °C evaporation was predicted to be 83 mm Hg absolute pressure, or -26.7 inches Hg gauge pressure.

The AZ-102 evaporator feed solution was evaporated on 5/16/00 in the evaporation apparatus as configured in Figure 1. Stainless steel wool, a vigreux column and de-entrainment finger were in place and 0.5 grams of antifoam chemical (DowCorning 1520-US) was added to the evaporator feed solution. A detailed account of the evaporation parameters is presented in Table 4. Approximately 305 mL of AZ-102 concentrate was produced.

Table 2. Analyses of AZ-102 Evaporator Feed (Decontaminated Envelope B Product)

Sample ID		AZ-102-Prod-1		AZ-102-Prod-2		avg. duplicate
Cs-137 (uCi/mL)		0.664		0.631		0.647
Co-60 (uCi/mL)	<	0.002	<	0.001	<	0.002
Eu-154 (uCi/mL)	<	0.007	<	0.007	<	0.007
Eu-155 (uCi/mL)	<	0.009	<	0.009	<	0.009
Sb-125 (uCi/mL)	<	0.019	<	0.020	<	0.020
Tc-99 (ug/L)		0.006		0.009		0.008
Pu-238 (dpm/mL)		507.46		464.52		485.99
Pu-239/240 (dpm/mL)		1011.61		1086.51		1049.06
mass 230 (ug/L)	<	0.095	<	0.095	<	0.095
231	<	0.095	<	0.095	<	0.095
232		57.91		16.67		37.29
233	<	0.095	<	0.095	<	0.095
234	<	0.095	<	0.095	<	0.095
235		25.56		29.60		27.58
236	<	0.095	<	0.095	<	0.095
237		126.23		146.18		136.20
238		2371.11		2305.96		2338.54
239	<	0.095	<	0.005	<	0.050
240	<	0.095	<	0.005	<	0.050
241	<	0.095	<	0.005	<	0.050
242	<	0.095	<	0.005	<	0.050
243	<	0.095	<	0.005	<	0.050
244	<	0.095	<	0.005	<	0.050
245	<	0.095	<	0.005	<	0.050
246	<	0.095	<	0.005	<	0.050
U - (mg/L) by Chem check		1.59		1.63		1.61
AA (mg/L)						
K		2679.20		2488.66		2583.93
Na		51648.02		50127.63		50887.83
As		0.318		0.340		0.329
Se		0.407		0.406		0.407
Hg	<	0.110	<	0.112	<	0.111
Carbon (mg/L)						
inorganic (TIC)		7998.03		9234.18		8616.11
Organic (TOC)		11638.51		5050.12		8344.32
density (g/mL)		1.098		1.095		1.097
wt.% Total solids		12.44		12.59		12.52
wt.% suspended solids		0.008	<	0.001	<	0.004
ALO ₂ (M)	<	0.020	<	0.020	<	0.020
Free OH (M)		0.224		0.222		0.223
IC (mg/L)						
NO ₃ ⁻		13447.72		13328.27		13388.00
NO ₂ ⁻		26211.48		26082.91		26147.20
PO ₄		242.70		247.44		245.07
SO ₄		13403.60		13170.80		13287.20
Oxalate		2382.86		2418.21		2400.54
Formate	<	110.32	<	112.47	<	111.40
Cl- (by IC)		66.19		67.48		66.84
F- (by IC)		860.48		877.30		868.89
Cl- (by ISE)		154.44		209.60		182.02
F- (by ISE)		860.48		866.06		863.27
ICP-E.S (mg/L)						
Al		407.67		396.02		401.85
B		1.15		1.12		1.14
Ba		1.35		1.34		1.34
Ca		0.68		0.45		0.57
Cd	<	0.07	<	0.07	<	0.07
Co		0.12		0.16		0.14
Cr		534.96		530.40		532.68
Cu	<	0.07	<	0.07	<	0.07
Fe	<	0.07	<	0.07	<	0.07
La	<	0.24	<	0.25	<	0.25
Li	<	0.04	<	0.04	<	0.04
Mg	<	0.02	<	0.02	<	0.02
Mn	<	0.02	<	0.02	<	0.02
Mo		43.13		43.20		43.17
Na		41985.73		40489.74		41237.73
Ni	<	0.15	<	0.16	<	0.16
P		137.56		141.27		139.41
Pb		0.99		1.35		1.17
Si		86.85		112.50		99.68
Sn		6.86		8.12		7.49
Sr	<	0.02	<	0.02	<	0.02
Ti	<	0.04	<	0.04	<	0.04
V		0.35		0.37		0.36
Zn		0.46		0.40		0.43

Table 3. Active AZ-102 Evaporation Calculations to Determine Water Removal Based on OLI Calculations

	Feed	Product	Condensate
Salt	102.80 g	102.80 g	0.00 g
Water	719.58 g	374.18 g	-345.40 g
Total	822.38 g	476.98 g	-345.40 g
Density	1.097 g	1.179 g	
Volume	750.0 mL	404.60 mL	
% solids	12.50%	21.55%	
% water	87.50%	78.45%	
% water removed		48.00%	

Antifoam Required, mg/kg	832.6
Active Antifoam Concentration, wt %	100.0%
Antifoam solution to add, g	0.68 g

Boilup rate			60.00 g/hr
Kettle Diameter			3.5 inches
Flux, g/cm ² -hr			0.06 g/hr-cm ²
Flux, lb/ft ² -hr			1.98 lb/hr-ft ²

Notes:

Start with 750 mL of AZ102 Env. B

Measured Total Dissolved Solids = 12.5 wt%

Measured Density = 1.0965

OLI Model suggest removal of 48 wt% water from AZ102 to attain 80% saturation at 25°C; Conclude that ~ 345 mL of water should be collected as condensate; Estimated concentration factor = $750/404.6 = 1.85$

Table 4. Active AZ-102 Evaporation Data

Time	T3 (°C)	T1 (°C)	T2 (°C)	P1 Press. (inches Hg)	Pot Vol. (mL)	Condensate Vol. (mL)	T4 (°C)	Volts* (V1)	Amps* (I1)
07:35	19.5	18.5	19.2	26.8	550	0	18.8	Off	Off
07:45	19.9	18.7	19.3	26.8	550	0	19.4	40	1.56
08:00	20.4	44.6	41.7	27.2	550	0	19.7	40	1.55
08:30	25.4	47.6	45.8	27.2	530	20	19.9	40	1.54
09:00	26.5	47.8	46.1	27.4	525	25	20.3	40	Nm
09:30	27.5	48.5	46.8	27.4	500	50	20.6	40	1.53
10:00	26.7	49.0	46.7	27.4	450	80	20.7	40	1.54
10:30	25.4	49.3	46.7	27.4	410	110	20.3	40	1.55
11:00	26.1	49.1	46.7	27.4	410	140	20.3	40	1.55
12:00	26.2	48.7	47.0	27.4	380	170	20.6	40	Nm
12:30**	25.9	49.5	47.3	27.4	360	190	20.8	40	Nm
15:00	22.0	38.3	35.0	26.8	410	190	21.0	40	Nm
15:30	24.9	48.8	46.9	26.8	400	200	20.9	40	Nm
16:30	24.9	49.2	47.4	27.2	355	245	20.8	40	1.53
17:00	24.8	49.7	47.5	27.0	305	295	21.1	40	Nm

Notes:

* Nominal heat flux of 4" height x 3/8" outer diameter (30 cm²) heated zone of heat rod equal to ~ 2.9 W/cm²

V1/I1 = Variable voltage and current input to heating rod

P1 = Vacuum pressure gauge on Concentrate Pot

T1 = Type K thermocouple temperature measurement in Concentrate Pot

T2 = RTD temperature readout in Concentrate Pot

T3, T4 = RTD exit temperature measuring devices for primary and secondary condensers, respectively

Nm = not measured

** After 12:30 the evaporation testing was stopped and the sample was allowed to cool. Remaining AZ-102 evaporator feed was then added and the evaporation was started up again at 15:00

The concentrated AZ-102 and the condensate liquids were sampled within days after the evaporation experiment was completed. Analytical methods detailed in Table 1 were performed by the Analytical Development Section (ADS) of SRTC. Both the AZ-102 concentrate and condensate liquids were sampled in duplicate. Concentrate samples were volumetrically diluted 1:10 in DI water before submission for analyses. Analytical results were then corrected for this 10X-dilution factor. Data from the AZ-102 concentrate and condensate analyses are presented in Tables 5 and 6, respectively.

Mass spectrometry values for Tc-99 in units of mg/L were converted to specific activity values in units of μCi/mL via Equation 1,

Equation 1:

$$a = 3.5778E+05 \times (g / (t_Y \times M))$$

Where:

a = activity in Curies (Ci)

3.5778E+05 = constant (Ci*years/g)

g = mass in grams

$t_Y = \frac{1}{2}$ life of Tc-99 in years = 2.13E+05 years

M = mass number of Tc-99 = 99

Using the average mass spectral value for Tc-99 from Table 5 as 6 µg/L and solving Equation 1 above, one calculates an average specific activity for Tc-99 in the composite to be 1.0E-04 µCi/mL, via:

$$a(\text{Ci/L}) = 3.5778E+05 \times (0.000006 \text{ g/L} / (2.13E+05 \times 99))$$

$$a(\text{Ci/L}) = 1E-07 \text{ Ci/L, or } 1E-04 \text{ µCi/mL}$$

Two different calculations were performed with the average values of the data shown in Tables 5 and 6. First, the concentration factors calculated from the evaporation process were obtained by dividing the final concentrate average concentrations by the initial feed concentrations. These data are shown in Table 7. The average concentration factor from data shown in Table 7 is 1.9 +/- 0.2, which is indeed close to the estimated target concentration factor determined from the evaporation parameters shown earlier in Table 3 to be ~ 1.85.

A second calculation was performed to determine the decontamination factor (DF) from the evaporation process. The evaporator feed solution concentration values were divided by the average condensate concentration values to determine how well the resulting condensate was decontaminated. These data are shown in Table 8. Comparison of the Cs-137 values indicate a DF of > 2E+05. Atomic absorption data for Na and K indicate a DF of > 2E+04. Ion chromatography data show a DF of > 1E+03. However, the relatively high reported detection limits (2 ppm to 10 ppm) for the condensates analyzed by IC influence this DF. Emission Spectroscopy data show DF's of > 7E+04.

It is possible to estimate an upper limit for the ratios of the gamma emitters to sodium for both Table 2 evaporator feed and Table 5 evaporator concentrate. For instance, one can divide the mda values for the gamma emitters (Co-60, Eu-154, Eu-155 and Sb-125) by the sodium value in Table 2 to get ratios that are all 'less than' values since the gamma emitters were all reported as less than values. Since the mda values for these same gamma emitters from Table 5 evaporator concentrate were nominally the same and the sodium concentration was increase by ~ 1.9X, then similar gamma emitter to sodium

radios for Table 5 will be reduced by $\sim 1.9X$. As example, from Table 2 the ratio of Co-60 to sodium is $<0.002 \mu\text{Ci/mL}$ (41,000 to 50,000 mg/L sodium). The same ratio calculated from Table 5 evaporator concentrate data is $<0.002 \mu\text{Ci/mL}$ (89,000 to 100,000 mg/L sodium). The above calculations are possible because of the quantifiable amount of sodium in the matrix. One can not obtain any information on the ratios of the mda gamma emitters from either Table 2 or Table 5 because all values are reported as less than values, i.e., from Table 2 the ratio of Co-60 ($<0.002 \mu\text{Ci/mL}$) to Eu-154 ($<0.007 \mu\text{Ci/mL}$) is indeterminate because both values are upper limit instrument detection values. Similarly for Table 5, the ratio of Co-60 ($<0.002 \mu\text{Ci/mL}$) to Eu-154 ($<0.006 \mu\text{Ci/mL}$) is indeterminate.

Table 5. AZ-102 Concentrate Analyses

		Env. B, AZ-102		Env. B, AZ-102		Env. B, AZ-102	
		Concentrate		Concentrate		Average	
		Sample #1		Sample #2		Average	
Methods/Analytes							
Gamma PHA							
Cs-137	μCi/mL		1.21E+00		1.26E+00		1.23E+00
Co-60		<	2.00E-03	<	2.25E-03	<	2.13E-03
Eu-154		<	6.16E-03	<	6.25E-03	<	6.21E-03
Eu-155		<	9.43E-03	<	8.41E-03	<	8.92E-03
Sb-125		<	2.68E-02	<	2.70E-02	<	2.69E-02
Separate/BetaCount							
Tc-99		<	1.00E-02	<	8.74E-03	<	9.39E-03
Sr-90	μCi/mL		1.13E+00		1.35E+00		1.24E+00
ICP-MS							
Tc-99	μg/L		6.2		5.7		6.0
Calculated Tc-99	μCi/mL		1.1E-04		9.7E-05		1.0E-04
mass 230	μg/L	<	0.65	<	0.65	<	0.65
231		<	0.65	<	0.65	<	0.65
232		<	0.65	<	0.65	<	0.65
233		<	0.65	<	0.65	<	0.65
234		<	0.65	<	0.65	<	0.65
235			33.9		35.9		34.90
236		<	0.65	<	0.65	<	0.65
237			200.0		199.9		199.95
238			3830.0		3820.0		3825.00
239			106.3		44.0		75.15
240		<	0.65	<	0.65	<	0.65
241		<	0.65	<	0.65	<	0.65
242		<	0.65	<	0.65	<	0.65
243		<	0.65	<	0.65	<	0.65
244		<	0.65	<	0.65	<	0.65
245		<	0.65	<	0.65	<	0.65
246		<	0.65	<	0.65	<	0.65
AA							
K	mg/L		4771		4339		4555
Na			102838		98796		100817
Inorganic (TIC)	mg/L		9684		9108		9396
Organic (TOC)			14576		25252		19914
density (g/mL)			1.158		1.164		1.161
wt% Total solids	wt%		25.6		24.3		25.0
AlO ₂ (M)	M	<	0.2	<	0.2	<	0.2
Free OH (M)			1.0		1.1		1.1
CO ₃ (M)			0.5		0.5		0.5

Table 5, Continued			Env. B, AZ-102		Env. B, AZ-102		
			Concentrate		Concentrate		Env. B, AZ-102
			Sample #1		Sample #2		Average
IC - Anions							
NO ₃	mg/L		27590		27020		27305
NO ₂			49900		48980		49440
PO ₄		<	1000	<	1000	<	1000
SO ₄			26550		26050		26300
Oxalate			4650		4600		4625
Formate		<	1000	<	1000	<	1000
Cl		<	200	<	200	<	200
F			1390		1350		1370
ICP-ES							
Al	mg/L		798.1		797.1		797.6
B		<	4.6	<	4.6	<	4.6
Ba		<	1.8	<	1.8	<	1.8
Ca			59.6		74.3		67.0
Cd		<	2.7	<	2.7	<	2.7
Ce		<	286.7	<	286.7	<	286.7
Co		<	4.6	<	4.6	<	4.6
Cr			1018.5		1017.7		1018.1
Cu		<	2.7	<	2.7	<	2.7
Fe		<	5.5	<	5.5	<	5.5
La		<	10.0	<	10.0	<	10.0
Li		<	1.8	<	1.8	<	1.8
Mg			2.1		4.0		3.1
Mn		<	0.9	<	0.9	<	0.9
Mo			77.3		80.9		79.1
Na			89249.4		89270.4		89259.9
Ni		<	6.4	<	6.4	<	6.4
P			228.7		230.5		229.6
Pb			25.7		25.5		25.6
Si			59.8		52.4		56.1
Sn			16.5		17.5		17.0
Sr		<	0.9	<	0.9	<	0.9
Ti		<	1.8	<	1.8	<	1.8
V		<	2.7	<	2.7	<	2.7
Zn		<	2.7	<	2.7	<	2.7
Zr		<	3.6	<	3.6	<	3.6

Table 6. AZ-102 Condensate Analyses

		Env. B, AZ-102		Env. B, AZ-102		Env. B, AZ-102	
		Condensate		Condensate		Average	
		Sample #1		Sample #2			
Methods/Analytes							
Gamma PHA							
Cs-137	μCi/mL	<	2.70E-06	<	2.61E-06	<	2.66E-06
Co-60		<	2.25E-06	<	1.81E-06	<	2.03E-06
Eu-154		<	2.27E-06	<	1.32E-05	<	7.74E-06
Eu-155		<	3.25E-06	<	2.15E-06	<	2.70E-06
Separate/BetaCount							
Tc-99	μCi/mL	<	4.77E-05	<	9.23E-05	<	7.00E-05
Sr-90		<	2.78E-05	<	2.78E-05	<	2.78E-05
ICP-MS							
Tc-99	μg/L	<	0.003	<	0.003	<	0.003
Calculated Tc-99	μCi/mL	<	5.26E-08	<	5.26E-08	<	5.3E-08
mass 230	μg/L	<	0.10	<	0.10	<	0.10
231		<	0.10	<	0.10	<	0.10
232		<	0.10	<	0.10	<	0.10
233		<	0.10	<	0.10	<	0.10
234		<	0.10	<	0.10	<	0.10
235		<	0.10	<	0.10	<	0.10
236		<	0.10	<	0.10	<	0.10
237		<	0.10	<	0.10	<	0.10
238		<	0.10	<	0.10	<	0.10
239		<	0.10	<	0.10	<	0.10
240		<	0.10	<	0.10	<	0.10
241		<	0.10	<	0.10	<	0.10
242		<	0.10	<	0.10	<	0.10
243		<	0.10	<	0.10	<	0.10
244		<	0.10	<	0.10	<	0.10
245		<	0.10	<	0.10	<	0.10
246		<	0.10	<	0.10	<	0.10
AA							
K	mg/L	<	0.135	<	0.135		0.135
Na			0.770		0.800		0.785
Inorganic (TIC)							
	mg / L		1.2		1.2		1.2
Organic (TOC)							
	mg / L		14.1		10.2		12.2
density (g/mL)							
			0.994		0.995		0.995
wt% Total solids							
	wt%	<	0.01	<	0.01		0.01
			Env. B, AZ-102		Env. B, AZ-102		

Table 6, Continued			Condensate		Condensate		Env. B, AZ-102
			Sample #1		Sample #2		Average
AlO ₂ (M)	Molar	<	0.002	<	0.002	<	0.002
Free OH (M)		<	0.002	<	0.002	<	0.002
CO ₃ (M)		<	0.002	<	0.002	<	0.002
IC - Anions							
NO ₃	mg/L	<	10	<	10	<	10
NO ₂		<	10	<	10	<	10
PO ₄		<	10	<	10	<	10
SO ₄		<	5	<	5	<	5
Oxalate		<	10	<	10	<	10
Formate		<	10	<	10	<	10
Cl		<	2	<	2	<	2
F		<	2	<	2	<	2
ICP-ES							
Al	mg/L		0.029		0.031		0.030
B			0.172		0.171		0.172
Ba		<	0.002	<	0.002	<	0.002
Ca			0.134		0.073		0.104
Cd		<	0.003	<	0.003	<	0.003
Ce		<	0.315	<	0.315	<	0.315
Co		<	0.005	<	0.005	<	0.005
Cr		<	0.007	<	0.007	<	0.007
Cu		<	0.003	<	0.003	<	0.003
Fe			0.007		0.006		0.007
La		<	0.011	<	0.011	<	0.011
Li		<	0.002	<	0.002	<	0.002
Mg			0.011		0.007		0.009
Mn		<	0.001	<	0.001	<	0.001
Mo		<	0.006	<	0.006	<	0.006
Na			0.851		0.836		0.844
Ni		<	0.007	<	0.008	<	0.008
P		<	0.026	<	0.026	<	0.026
Pb		<	0.028	<	0.028	<	0.028
Si			1.172		1.174		1.173
Sn			0.016		0.015		0.016
Sr		<	0.001	<	0.001	<	0.001
Ti		<	0.002	<	0.002	<	0.002
V		<	0.003	<	0.003	<	0.003
Zn		<	0.003	<	0.003	<	0.003
Zr		<	0.004	<	0.004	<	0.004

Table 7. Concentrations Factors Calculated from Evaporation of AZ-102

Envelope B, AZ 102					
		Evaporator Feed		Concentrate	Concentration Factor
Gamma PHA					
Cs-137	(μ Ci/mL)	0.647		1.23E+00	1.9
AA	(mg/L)				
K		2584		4555	1.8
Na		50888		100817	2.0
IC	(mg/L)				
NO ₃		13388		27305	2.0
NO ₂		26147		49440	1.9
PO ₄		245	<	1000	
SO ₄		13287		26300	2.0
Oxalate		2401		4625	1.9
Formate		< 111	<	1000	
Cl		67	<	200	
F		869		1370	1.6
ICP-ES	(mg/L)				
Al		401.8		797.6	2.0
B		1.1	<	4.6	
Ba		1.3	<	1.8	
Ca		0.6		67.0	
Co		0.1	<	4.6	
Cr		532.7		1018.1	1.9
Mg		< 0.01		3.1	
Mo		43.2		79.1	1.8
Na		41237.7		89259.9	2.2
P		139.4		229.6	1.6
Pb		1.2	<	25.6	
Si		99.7		56.1	
Sn		7.5		17.0	2.3
V		0.4	<	2.7	
Zn		0.4	<	2.7	

Table 8. Decontamination Factors Calculated from Evaporation of AZ-102

		Env. B, AZ-102		Env. B, AZ-102		
		Feed		Condensate		Decontamination Factor
		Average		Average		
Gamma PHA						
Cs-137	μCi/mL	0.6474	<	2.66E-06	>	2.4E+05
AA	(mg/L)					
K		2584	<	0.135	>	1.9E+04
Na		50888		0.785		6.5E+04
IC	(mg/L)					
NO3-		13388	<	10	>	1.3E+03
NO2-		26147	<	10	>	2.6E+03
PO4		245	<	10	>	2.5E+01
SO4		13287	<	5	>	2.7E+03
Oxalate		2401	<	10	>	2.4E+02
Cl		67	<	2	>	3.3E+01
F		869	<	2	>	4.3E+02
ICP-ES	(mg/L)					
Al		401		0.030		1.3E+04
Cr		532	<	0.007	>	7.6E+04
Mo		43.2	<	0.006	>	7.2E+03
Na		41237		0.844		4.9E+04
P		139	<	0.026	>	5.3E+03

Waste Glass Formulation

Results of the initial AZ-102 evaporator feed previously shown in Table 2 were transmitted to the VSL to develop a glass formulation for the waste stream. The evaporator feed supernate was submitted for glass formulation instead of the concentrated evaporator product supernate in the interest of obtaining the glass formulation early. Table 9 shows the AZ-102 glass formulation that was based on a sodium molarity of 2.21 Molar. The glass formulation shown in Table 9 that was based on the evaporator feed data shown in Table 2 was subsequently reexamined by VSL personnel after the evaporator concentrate characterization became available (Table 5 data). VSL personnel confirmed that no significant changes to the glass formulation were required.

In the past, glass former formulations received from VSL for previous LAW glasses for Hanford tanks Env. A (AN-103) and Env. C (AN-102), have included sugar as a reductant [11,12]. The glass formulation shown in Table 9 for this Env. B AZ-102 LAW glass uses a mixture of ten different minerals. Note that no added sugar was needed due to the measured 8 g/L levels of soluble total organic carbon in the sample. The target waste loading for this AZ-102 glass is nominally 5 wt% Na₂O and 0.8 wt% SO₃. Concurrence with this particular glass recipe was also obtained from RPP-WTP. The recipe shows that ten different minerals are used as the glass former mixture. The recipe shows that for every one mole of sodium from pretreated feed, a target of 620 grams of glass is made from adding the feed to 691 grams of glass former mixture.

The ratio of sulfate to sodium in the AZ-102 waste is significantly higher than the previous two LAW tank wastes studied at SRTC. Appendix B shows data for the three LAW tank waste glass formulations studied. Comparison of Env. A (AN-103), Env. C (AN-102) and Env. B. (AZ-102) shows that for the lower concentrated sulfate stream (Env. A, AN-103), a waste loading of 20 wt% Na₂O could be achieved with a target sulfur loading at 0.1 wt% SO₃. As the sulfate concentration increased for the Env. C, AN-102 sample to 0.4 wt% SO₃, the target waste loading decreased down to 12 wt% Na₂O. For the highest target sulfate concentration of 0.8 wt% SO₃ in Env. B, AZ-102 the target waste loading was only 5 wt% Na₂O.

Glass Former Blend Preparation and Analysis

A batch of glass formers was prepared for this study using the recipe for the AZ-102 glass supplied by VSL. The blend of glass formers identified as 9382-130 was weighed on a calibrated balance and blended with an automatic shaker for ten minutes. The blends were split into smaller representative samples with a Quantachrome Sieving Rotary Riffler. The riffler device consists of a central container that when filled, is shaken at a high frequency causing the contents to be slowly emptied into a rotating tray of eight equal sized stainless steel pans. Splitting a mixture with the riffler device ensures that resulting portions are representative of the starting batch of material. The glass former blend mixtures were split into 1/8th samples and seven samples were

recombined. One, 1/8th sample was split into 1/64th samples. Four of the 1/64th samples were combined and split into eight parts or 1/128th samples for ADS chemical analysis by ICP-ES. The intent of this analysis was to simply confirm that the mixture of glass former minerals agreed with target for the ten major elements added from the glass formers. The glass former blend was made according to data shown in Table 10. The last data row highlighted in Table 10 indicates that analyzed values agree well with the target blend, with analyzed values within ~ 8% of target for the ten major elements from the glass former minerals. Limited data on Universal Treatment Standard (UTS) RCRA metals were obtained from the ICP-ES data. Elemental wt% values for Ba, Cr, Cd, Pb and V are shown. Other UTS RCRA metals (As, Se, Hg, Ag, Sb, Be and Tl) were not analyzed in these analyses by ICP-ES. The UTS RCRA minerals Ni and Zn were also measured. Nickel derives from the nickel crucibles used in the peroxide fusion and thus the analyzed value can not be used. Zinc is added as glass former component from zinc oxide. More detailed information on the glass formers and the various RCRA metals content can be found in Reference # 10.

Table 9. AZ-102 Glass Formulation from Vitreous State Laboratory

Recipe for glass LAWB53 using AZ102 High SO3 active waste														21-Jun-00	
Envelope Constituents	calculated M	Evapor. feed AZ-102 mg/L	GLASS AZ102 Oxides	AZ102 wt% As glass	AZ102 wt% in glass @ 5% Na2O	Glass Former Mix	LAWB53 this target for AZ102	Additives this sample	Source in Additives	Assay	Ratio	Target Weight (g)	other oxides present	Vendor Information	
		5/15/00 email	Loading	1.000	6.31%	100%	0.0631	93.69%				Additives	% Fe2O3	% SiO2	
Ag			Ag2O	0.000	0.0000		0.0000								
Al	0.01	402	Al2O3	0.878	0.055	6.45	6.0987	6.043	Kyanite (Al2SiO5) 325 Mesh	0.990	0.540	70.04	1.16%	43.70%	Kyanite Mining
B		1.1	B2O3	0.004	0.0003	10.70	10.0256	10.025	H3BO3 (Technical Granular)	0.986	0.563	111.90			US Borax
Ba		1	BaO	0.002	0.0001		0.0001								
Ca		1	CaO	0.001	0.0001	7.20	6.7461	6.746	Wollastonite NYAD 325 Mesh	0.993	0.475	88.62	0.40%	51.00%	NYCO Minerals
Cd		0.1	CdO	0.000	0.0000		0.0000								
Co		0.1	CoO	0.000	0.0000		0.0000								
Cr		532.7	Cr2O3	1.800	0.1135		0.1135								
Cu		0.0	CuO	0.000	0.0000		0.0000								
Fe		0.0	Fe2O3	0.000	0.0000	5.70	5.3406	5.341	Fe2O3 (Iron III oxide, -325 Mesh)	0.998	1.000	28.96			Alfa Aesar-Johnson Matthey
K	0.07	2584	K2O	3.598	0.227		0.2269								
La		0	La2O3	0.000	0.0000		0.0000								
Li		0	Li2O	0.000	0.0000	6.25	5.8559	5.856	Li2CO3 (Chemetal Foote Co. Tech. gr.)	0.99	0.404	90.72			Cyprus Foote Mineral Co.
Mg		0	MgO	0.000	0.0000	3.20	2.9982	2.998	Olivine (Mg2SiO4) 325 Mesh (#180)	0.990	0.480	39.09	7.68%	42.52%	UNIMIN Corp.
Mn		0	MnO2	0.000	0.0000		0.0000								
Mo		43.2	MoO3	0.075	0.0047		0.0047								
Na	2.21	50888	Na2O	79.298	5.00		5.0000								
Ni		0	NiO	0.000	0.0000		0.0000								
Pb		1.2	PbO	0.001	0.0001		0.0001								
Sn		0	SnO2	0.000	0.0000		0.0000								
Si		100	SiO2	0.246	0.0155	52.20	48.9242	48.909	SiO2 (Sil-co-Sil 75)	0.997	1.000	202.07			US SILICA
Sr		0	SrO	0.000	0.0000		0.0000								
Ti		0	TiO2	0.000	0.0000	1.50	1.4054	1.405	TiO2 (Rutile Airfloated)	0.954	1.000	9.13	0.71%	0.91%	Chemalloy
W		0	WO3	0.000	0.0000		0.0000								
Zn		0.4	ZnO	0.001	0.0000	3.40	3.1857	3.186	ZnO (Kadox-920)	0.997	1.000	19.80			Zinc Corp. of America
Zr		0	ZrO2	0.000	0.0000	3.40	3.1856	3.186	Zircon ZrSiO4 (Flour) Mesh 325	0.990	0.651	30.63		33.00%	American Mineral
Br		0	Br	0.000	0.0000		0.0000								
Cl		67	Cl	0.077	0.0049		0.0049								
F		869	F	1.004	0.0633		0.0633								
PO4		245	P2O5	0.212	0.0133		0.0133		Total Sodium Moles			1.00 moles			
SO4	138.3E-3	13287	SO3	12.802	0.8072		0.8072		Expected Glass yield			620 g			
NO2	0.57	26147	NO2						Sum of Additives (g)			691 g			
NO3	0.22	13388	NO3						Sugar as added reductant (decreased for TOC)			0 g			
OH			OH												
CO3	0.14	8616	CO3												
oxalate		2400	C												
formate			C												
SUM			SUM	100.000	6.31	100.00	100.00	93.695							
NO2+NO3 TOC	0.78 MI		VSL uses 12 moles Carbon (1 mole sucrose/342.3g) per 16 Moles NOx in order to mitigate foaming.												
		8344.3 mg/L	This feed requires 7.059 g/L Carbon. This sample already includes sufficient TOC												

Table 10. Glass Former Blend NB#9382-130, Prepared and Analyzed at SRTC

	Glass LAZ102, 9382-130			RFS 6/28/00							B Env.												
	Batch Wt	SiO2	Al2O3	Fe2O3	B2O3	BaO	Cr2O3	CaO	CdO	MgO	ZnO	NiO	Li2O	Na2O	PbO	K2O	TiO2	ZrO2	V2O5	MnO	SO4	Total	
Glass Former	grams																						
Isabelle's GF Mix		54.00	6.18	7.50	11.70			7.40		1.75	3.50		3.17				1.30	3.50					100.00
Kyanite Raw	70.04	30.61	37.82	0.81				0.02		0.01				0.29			0.76						70.32
Boric Acid	111.90	0.00			63.34																		63.34
Wollastonite-NYAD325	88.62	45.20	0.18	0.35				42.09		0.09							0.02			0.09	0.01		88.02
Fe2O3, Alfa	28.96	0.00		28.96																			28.96
Li2CO3 Cyprus	90.72							0.05					36.39	0.08		0.03						0.15	36.55
Olivine, Unimin-180	39.09	16.62	0.07	3.00			0.05	0.01		18.77		0.14		0.01		0.00							38.68
Silica, SCS75	202.07	201.46	0.18	0.04				0.08									0.02						201.79
TiO2, RutileAF	9.13	0.08		0.06			0.02										8.71	0.08	0.04		0.00		8.99
ZnO, Kadox 920	19.80	0.00		0.00					0.001		19.76				0.000								19.76
Zircon, flour AmMin	30.63	9.88	0.08	0.02													0.03	20.22					30.23
Sugar, FL	0.00																						0.00
Totals	690.96	303.85	38.33	33.26	63.34		0.07	42.25		18.86	19.76	0.14	36.39	0.38		0.04	9.54	20.30	0.04	0.09	0.16		586.63
Ele/Oxide		0.47	0.53	0.70	0.31		0.68	0.71		0.60	0.80	0.79	0.46	0.74		0.83	0.60	0.74	0.56	0.77	0.33		
Wt% Oxide		51.80	6.53	5.67	10.80		0.012	7.20		3.22	3.37	0.02	6.20	0.07		0.01	1.63	3.46	0.01	0.02	0.03		100.00
Wt% Element		24.22	3.46	3.97	3.35		0.008	5.15		1.94	2.70	0.02	2.88	0.05		0.01	0.97	2.56	0.00	0.01	0.01		51.31
Wt% Ox. /Batch	0.85	45.58	5.75	4.99	9.50		0.010	6.34		2.83	2.96	0.02	5.46	0.06		0.01	1.43	3.04	0.01	0.01	0.02		88.00
Wt% Ele./Batch	0.85	21.32	3.04	3.49	2.95		0.007	4.53		1.71	2.38	0.02	2.54	0.04		0.00	0.86	2.25	0.00	0.01	0.01		45.16
Wt% Ele. (Na2O2) ADS		20.30	2.82	3.45	2.72	0.004	0.040	4.28	0.008	1.70	2.40	(**)	2.48	(**)	0.051		0.85	2.12	0.011	0.18			43.23
%Diffbatch vs ADS		4.78	7.22	1.19	7.80			5.53		0.57	-0.84		2.15				0.73	5.97					

(**) Ni and Na used in the nickel crucibles by sodium peroxide fusion

Estimated Radionuclides in AZ-102 Glass

Radiochemical analyses data from the decontaminated feed AZ-102 evaporator concentrate material can be used along with the glass formulation recipe to estimate the expected level of radionuclides in the product glass. Three radionuclides are of specific interest per the Hanford RPP contract [1]. They are Cs-137, Sr-90 and Tc-99. Table 11 shows the estimated activities of these radionuclides in the product glass. The concentrations of each radionuclide are taken from Table 5 and the amount of supernate required is taken from the total moles of sodium (See Table 9, VSL Glass Formulation) required to make the specified amount of glass. Comparison of the expected specific activities for these radionuclides to the target upper limit values shown in the notes to Table 11 indicate that the target Env. B, AZ-102 glass should meet Hanford RPP contract specification limits. While the WTP Contract specifies an average Cs-137 concentration of less than 3 Ci/m³ in the ILAW glass, the WTP project has subsequently adopted a design basis that limits the Cs-137 concentration to less than 0.3 Ci/m³. This lower limit was adopted after the AZ-102 sample had been pretreated at SRTC.

Tables 2 and 5 also include radiochemical data and ICP-MS data for the actinides that can be used to estimate the transuranic (TRU) loading expected for the glass. Table 11 shows the concentrations for the actinides with mass number from 230 to 246 from ICP-MS analyses of the evaporator concentrate from Table 5. The instrument detection limit for these analyses was 0.065 µg/L. However, the samples were diluted 10X before submission, so the actual detection limit reported in Table 11 is 0.65 µg/L. Data from the evaporator feed Table 2 was used for Pu-238 since these values were from actual radiochemical analyses. Summation of all the actinide concentrations in glass (µCi/g) shows that total specific activity of these actinides totals to less than 64 ηCi/g, which is well below the TRU limit of 100 ηCi/g. Additionally, only some of the actinides shown in Table 11 are actual alpha-emitting TRU radionuclides, so the value of < 64 ηCi/g is a conservative upper bound on the TRU content expected in the glass.

The ICP-MS data (µg/L) were converted to specific activities in the glass (µCi/g) using Equation 1, via:

$$\begin{aligned} \mu\text{Ci/g glass} \\ = \text{Constant} \times (\mu\text{g/L} \times (.045\text{L}/111\text{g})) \times (1\text{g}/1\text{E}+06\mu\text{g}) / (t_{\gamma} \times M) \times (1\text{E}+06 \mu\text{Ci} / 1\text{Ci}) \end{aligned}$$

The radiochemical data for Pu-238 (disintegrations per minute per mL) were converted to specific activities in the glass (µCi/g) using Equation 1, via:

$$\begin{aligned} \mu\text{Ci/g glass} \\ = \text{Constant} \times (\text{dpm/mL} \times (1\text{m}/60\text{s}) \times (1000\text{mL}/\text{L}) \times (.045\text{L}/111\text{g})) \times (1\text{Ci}/3.7\text{E}+10 \text{dps}) / (t_{\gamma} \times M) \\ \times (1\text{E}+06 \mu\text{Ci}/1\text{Ci}) \end{aligned}$$

Table 11. Expected Average Radionuclide Specific Activities in AZ-102 Glass

Radionuclide	Analysis Method	Average Concentration (μCi/mL) (from Table 5)	Volume Liquid (mL)	Target Mass Glass (g)	Expected Specific Activity in Glass (μCi/g)	Expected Specific Activity in Glass [#] (Ci/m ³)
Cs-137	Gamma Pulse Height Analysis	1.23	43	110	0.48	1.3
Sr-90	Separation, Counting	1.24	43	110	0.48	1.3
Tc-99	ICP-MassSpec	1E-04	43	110	3.9-05	1.1E-04
Tc-99	Separation, Counting	<9.4E-03	43	110	<3.7E-03	<1.0E-02
ICP-MS						
Mass#	Half-Life	Concentrate		Concentrate		
		Sample 1		Sample 2		
	(years)	(μg/L)	μCi/g glass	(μg/L)	μCi/g glass	
mass 230 (Th)	7.54E+04	< 0.65	< 5.4E-06	< 0.65	< 5.4E-06	
mass 231(Pa)	3.28E+04	< 0.65	< 1.2E-05	< 0.65	< 1.2E-05	
mass 232 (Th)	1.40E+10	< 0.65	< 2.9E-11	< 0.65	< 2.9E-11	
mass 233 (U)	1.59E+05	< 0.65	< 2.5E-06	< 0.65	< 2.5E-06	
mass 234 (U)	2.45E+05	< 0.65	< 1.6E-06	< 0.65	< 1.6E-06	
mass 235 (U)	7.04E+08	3.39	3.0E-08	3.59	3.0E-08	
mass 236 (U)	2.34E+07	< 0.65	< 1.7E-08	< 0.65	< 1.7E-08	
mass 237 (Np)†	2.14E+06	20.00	5.7E-06	19.99	5.7E-06	
mass 238 (U)	4.47E+09	383.12	5.2E-08	382.60	5.2E-08	
mass 238 (Pu)†	8.77E+01		1.83E-07 ^{##}		1.68E-07 ^{##}	
mass 239 (Pu)†	2.41E+04	10.63	2.7E-04	4.40	2.7E-04	
mass 240 (Pu)†	6.56E+03	0.92	8.5E-05	< 0.65	< 8.5E-05	
mass 241 (Pu)	1.44E+01	< 0.65	< 2.7E-02	< 0.65	< 2.7E-02	
mass 241 (Am)†	4.33E+02	< 0.65	< 9.0E-04	< 0.65	< 9.0E-04	
mass 242 (Pu)†	3.76E+05	< 0.65	< 1.0E-06	< 0.65	< 1.0E-06	
mass 243 (Am)†	7.37E+03	< 0.65	< 5.3E-05	< 0.65	< 5.3E-05	
mass 243 (Cm)†	2.85E+01	< 0.65	< 1.4E-02	< 0.65	< 1.4E-02	
mass 244 (Cm)†	1.81E+01	< 0.65	< 2.1E-02	< 0.65	< 2.1E-02	
mass 245 (Cm)†	8.50E+03	< 0.65	< 4.5E-05	< 0.65	< 4.5E-05	
mass 246 (Cm)†	4.73E+03	< 0.65	< 8.1E-05	< 0.65	< 8.1E-05	
Sums		μCi/g	< 0.06		< 0.06	
Sums		ηCi/g	< 63.58		< 63.60	

Notes:

[#] Specification limits (Reference 1): < 3 Ci/m³ for Cs-137, < 20 Ci/m³ for Sr-90 and < 0.1 Ci/m³ for Tc-99; Estimated densities used for Env. B glass ~ 2.8 g/cm³.

^{##} Pu-238 values calculated from Evaporator Feed data from Table 2.

† These actinides are alpha-emitting transuranic radionuclides.

$$\mu\text{Ci/g glass} = \text{Constant} \times (\mu\text{g/L} \times (.045\text{L}/111\text{g})) \times (1\text{g}/1\text{E}+06\mu\text{g}) / (t_Y \times M)$$

Vitrification

Crucible-Scale Vitrification

Several crucible-scale vitrification experiments have been conducted during the course of this study. All vitrifications were performed in 600-mL Pt(95%)/Au(5%) crucibles obtained from ENGELHARD-CLAL company. Table 12 documents the details of all tests. Tests #1 - #4 were conducted on the AZ-102 radioactive concentrate material. Tests #5 and #6 were performed with VSL-supplied AZ-102 simulants. Test #5 used a dry powder blend (LAWB53SUR2) and Test #6 used a glass simulant (LAWB53SUR1) – both of these simulants were previously prepared at VSL. Tests #7 - #9 used a simulated AZ-102 liquid supernate recently developed at SRTC. All blends in Tests #1 - #4 and Tests #7 - #9 were formulated based on the characterization data for the AZ-102 supernate and the glass formulation recipe provided by VSL. The next to last column of Table 12 indicates whether Tests were performed with radioactive or simulant feeds. The crystal structure identification by x-ray diffraction analysis is included in the last column of Table 12. The empirical formulas of the different pyroxene crystals are shown at the bottom of Table 12. Temperature vs. time profiles for all crucible scale vitrification tests are presented in Appendix C. All of the details of all tests are described below in chronological order.

Table 12. Details of Crucible-Scale Vitrification Experiments

AZ-102 Vitrification Tests	Date	Furnace	Mass Glass Formers (g)	Volume Liquid (mL)	Target Glass Mass (g)	Notes	Crystal Structure Pattern Number And Crystal Identification
#1	7/5/00	Deltech	122.6	42.9	110	First of two replicate runs using radioactive AZ-102 , canister cooling	Not measured
#2	7/10/00	Deltech	122.6	42.9	110	Second replicate run with radioactive AZ-102	# 80-1862, unnamed clinopyroxene
#3	7/26/00	Deltech	122.6	42.9	110	Repeat of above two radioactive AZ-102 runs, with rapid cooling of glass	No crystals detected
#4	9/20/00	Deltech	(see text)	(see text)	100	Remelt of remaining radioactive AZ-102 glass from Test #3 above, canister cooling	# 80-1864, augite pyroxene
#5	10/30/00	Deltech	(see notes)	(see text)	100	Vitrification of VSL powder simulant , LAWB53SUR2, canister cool	# 80-1864, augite pyroxene
#6	11/6/00	Deltech	(see text)	(see text)	100	Vitrification of VSL glass simulant , LAWB53SUR1, canister cool	# 80-1864, augite pyroxene
#7	5/21/01	Deltech	122.6	43	110	Vitrification of SRTC-developed simulant , canister cooling	# 80-1864, augite pyroxene
#8	5/21/01	Thermolyne	122.6	43	110	Vitrification of SRTC-developed simulant in open crucible, canister cooling	# 80-1864, augite pyroxene
#9	6/11/01	Deltech (modified to allow higher air-flux)	122.6	40	110	Vitrification of SRTC-developed simulant in modified, higher air-flux sealed quartz Deltech furnace, canister cooling	# 80-1864, augite pyroxene

Empirical Formula and Oxidation States of Crystalline Pyroxenes (from Oberti and Caporuscio, 1991)

	M2 site				M1 site						T site	
	Ca ²⁺	Na ⁺	Fe ²⁺	Mg ²⁺	Mg ²⁺	Fe ²⁺	Fe ³⁺	Al ³⁺	Ti	Cr ³⁺	Si ⁴⁺	Al ³⁺
Unnamed Clinopyroxene	0.78	0.18	-	0.04	0.79	-	0.05	0.07	-	0.09	1.97	0.03
Augite	0.61	0.25	0.07	0.07	0.65	0.10	0.03	0.22	-	-	2	-

Vitrification Tests with Radioactive AZ-102 Supernate, Tests #1 - #4

The initial two radioactive glasses produced from AZ-102 (Tests #1 and #2) were not analyzed per SRTC Task Technical Plans [5,8] due to the observed secondary phase on the bottom of the glasses. Figure 4 shows these two glasses. Figure 4(A) is a digital photograph obtained while the glass in the Pt/Au crucible is being held inside of a radiochemical hood within the radiological buffer area lab in SRTC. It should be noted that all handling of the radioactive samples (feed and glass) in this study was conducted under the strict guidelines for radioactive work at SRTC. Routine radiation limits and proper personal protective equipment were in place. These radiation limits for working with radioactive samples in radiochemical hoods are <5 mrem/hr whole body dose and < 1,000 mrem/hr contact dose. Radiation workers are required to wear two sets of yellow-colored gloves and external dosimetry finger rings for anticipated contact doses > 50 mrem/hr.

Figures 4(B) and 4(C) show the bottom-formed crystals observed in the Test #1 and #2 glasses, i.e., the glass melt pucks have been turned upside down for the photograph. Both of these glasses were produced using ~ 53-hour evaporation and calcine heatup, a 4-hour melt, and allowed to cool via controlled ~ 56-hour simulated canister cooling. The presence of bottom-forming crystals on Test #2 glass confirmed that the observed crystals were indeed reproducible.

Figure 4. Photographs of Glasses Produced from Tests #1 and #2
4(A): Top Surface of Test #1 Glass Showing Dark Shiny Amorphous Surface with No Visible Crystalline Phase Present.

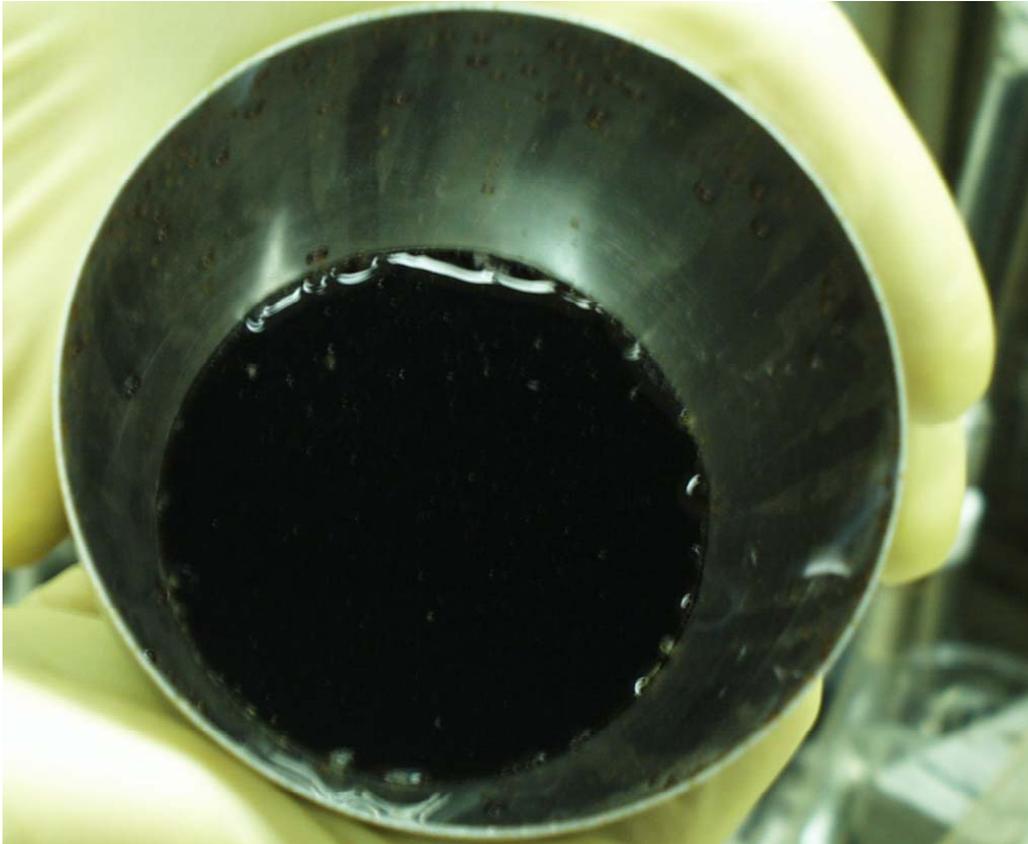


Figure 4(B): Bottom Surface of Test #1 Glass Showing Secondary Phase Crystals.
Note that the glass puck has been turned upside down to reveal the bottom surface.



Figure 4(C): Bottom Surface of Test #2 Glass Showing Secondary Phase Crystals.
Note that the glass puck has been turned upside down to reveal the bottom surface.



A third replicate glass was made (Test #3) with radioactive AZ-102 supernate. This glass was produced in similar manner to Tests #1 and #2 glasses, except that it was allowed to cool within about 3 hours to investigate the effects of relative rapid or quench cooling on the glass properties, i.e., complete cooling of the glass was accomplished within ~ 3 hours for Test #3 vs. the ~ 56-hour cooling used in Tests #1 and #2. This glass did not have a visible secondary phase on the bottom surface.

It was decided in discussions with Hanford RPP, VSL and SRTC personnel to pursue limited characterization of this Test #3 glass. The primary reason for this characterization was to establish that indeed the glass contained the same elemental composition as the target formulation. Agate mortar and pestle broke the Test #3 glass puck into small pieces. A few of the small pieces were pulverized to -200 mesh size using a Mixer Mill with agate cups and agate grinding balls. The ground glass was sieved through brass sieves to ensure a -200 mesh fraction was available for dissolution analysis and for XRD analysis.

X-ray diffraction analysis of the -200 mesh glass powder from this Test #3 glass indicated no presence of crystalline material as shown in Figure 5. The x-ray pattern shown in Figure 5 indicates that the scan was performed on an overnight scan, which is typically more accurate than the nominal scans performed over several hours time. Figures 6 – 8 show microscopy photographs of the +200 mesh size glass powders. These Scanning Electron Microscopy/Energy Dispersive x-ray data also indicate no presence of detectable crystalline structure in the glass fines. Some particles containing copper and platinum are visible and are likely due to use of brass sieves and the platinum crucibles, respectively.

Chemical analysis by ICP-ES of the dissolved -200 mesh glass agreed well with target glass formulation as shown in Table 13. Data shown in Table 13 for the Test #3 glass composition derives from sodium peroxide fusion of the glass in Ni-crucibles and from acid dissolution of the glass in polybottles. The boron value shown in Table 13 derives from sodium peroxide fusion data. The sodium value shown in Table 13 derives from ICP-ES analysis of the acid dissolution of the Test #3 glass. The target AZ-102 glass composition shown in Table 13 is taken from the VSL glass formulation shown previously in Table 9. The last column of Table 13 shows the average percent difference between the two dissolution methods and the target composition. Measured average values for iron and titanium were ~ 15% higher than target and measured sodium was ~ 10% lower than target. The sum of the measured oxides was in the range of 102 to 108 vs. the target 100%.

Conclusions from these first three vitrification Tests #1 - #3 were,

- (1) that the bottom-forming crystalline phase produced on the radioactive AZ-102 crucible melts formed only upon simulated canister cooling of the glass,
- (2) that the bottom-forming crystalline phase was reproducible, and
- (3) that the overall glass produced with rapid cooling met target composition for all the major elements to within the experimental analysis method of nominally 10% and did not contain a visible crystalline phase detectable by XRD analysis in the (-) 200 mesh ground glass sample.

Figure 5. XRD Pattern of Test #3 Glass Powder (-200 Mesh) Showing No Significant or Identifiable Crystalline Structure

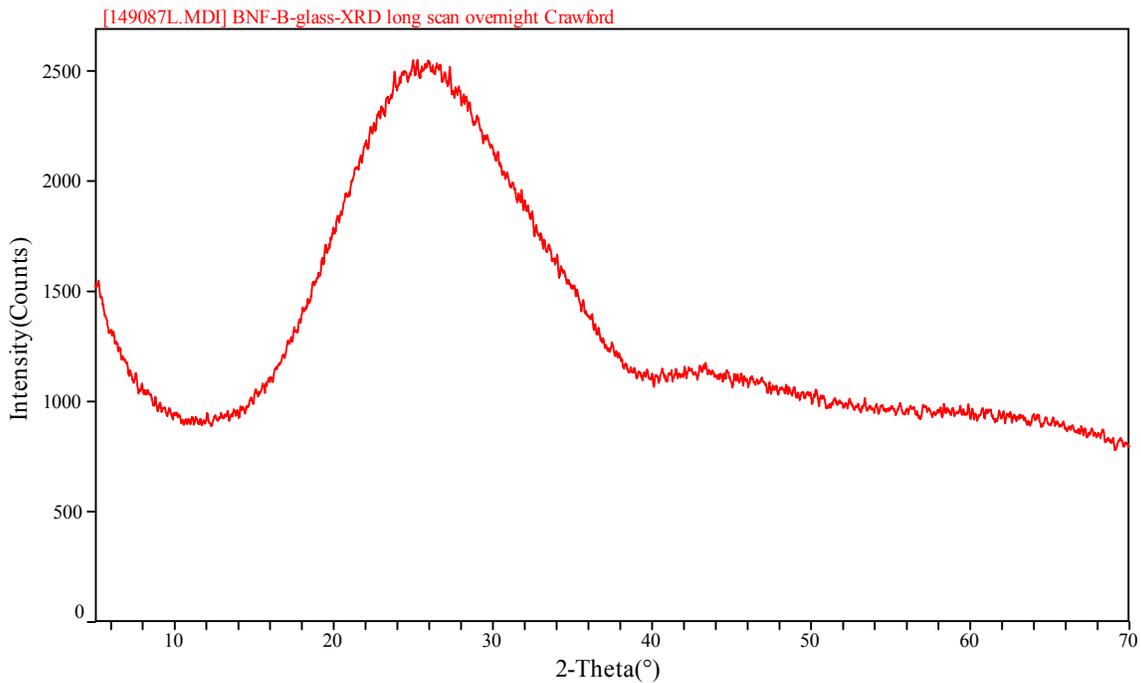


Figure 6. SEM Photos (100X Magnification) of the +200 Mesh Rapid-Cooled Glass
(A) Upper image is secondary electron image. This image shows numerous very small fines of glass covering the darker glass piece. This glass was not washed before SEM/EDAX analysis. No crystalline species are observed.

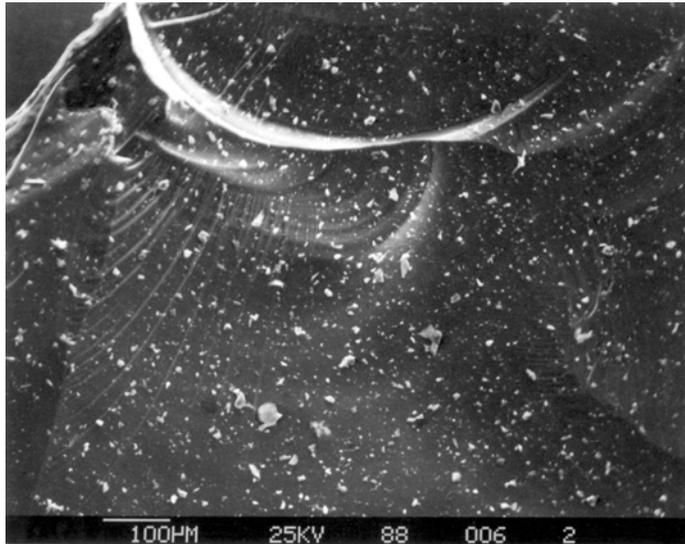


Figure 6(B) Lower image is backscattered replicate image of Figure 6(A).

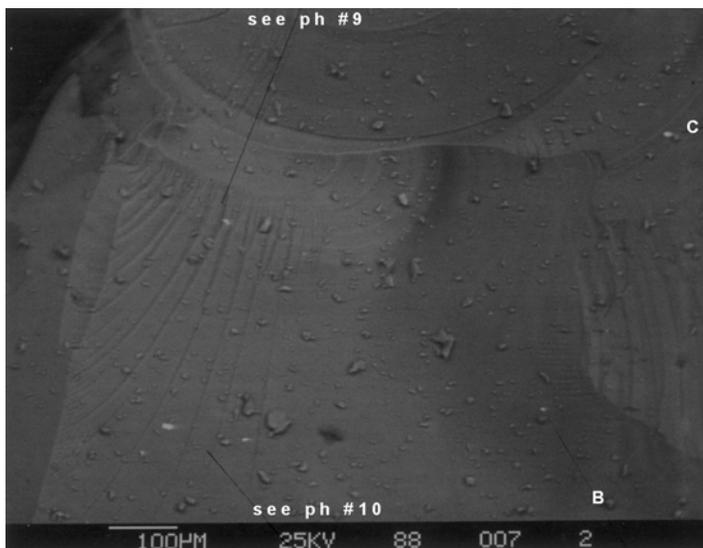


Figure 7. SEM Backscatter Photo of +200 Mesh Glass at 500X Magnification
7(A) Particles identified as 'A' and 'B' are shown by EDAX spectra to be:
7(B) Amorphous glass fines and
7(C) Likely copper from the brass sieve.

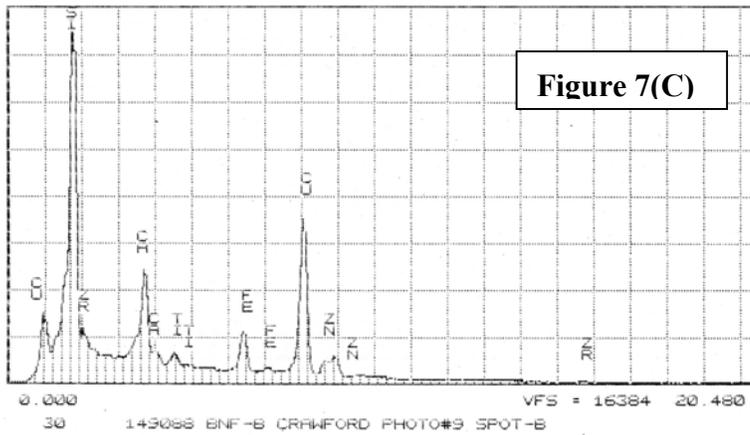
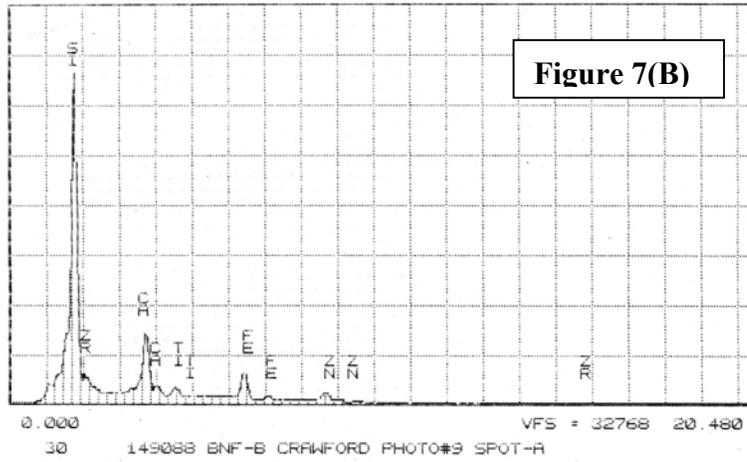
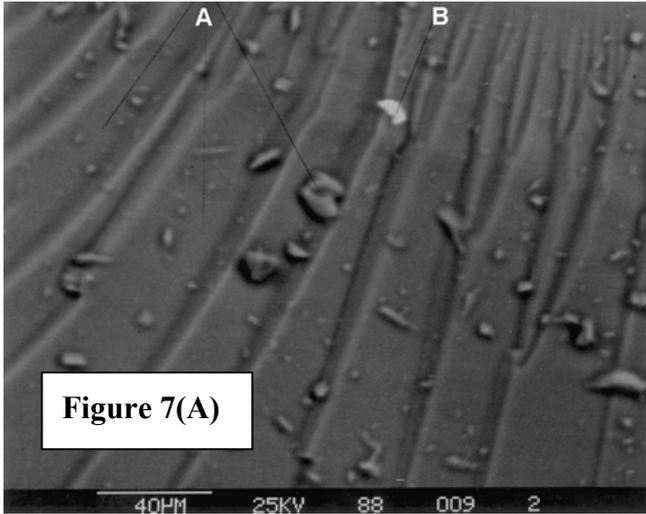


Figure 8. SEM Backscatter Photo of +200 Mesh Glass at 500X Magnification
8(A) Particles identified as 'C' and 'D' are shown by EDAX spectra to be:
8(B) Copper likely from the brass sieve and
8(C) Platinum from the platinum crucible

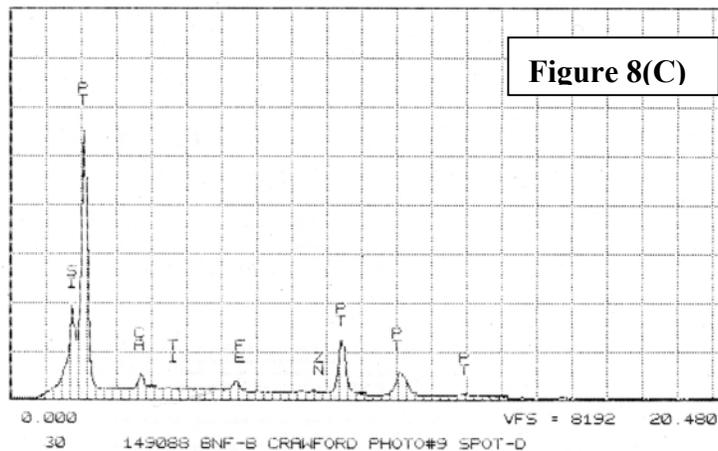
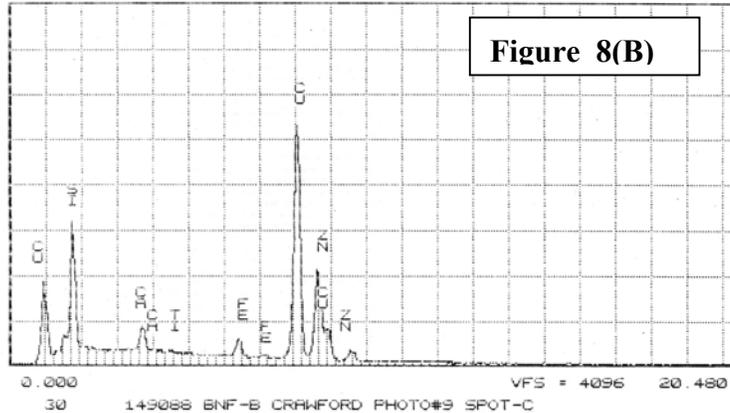
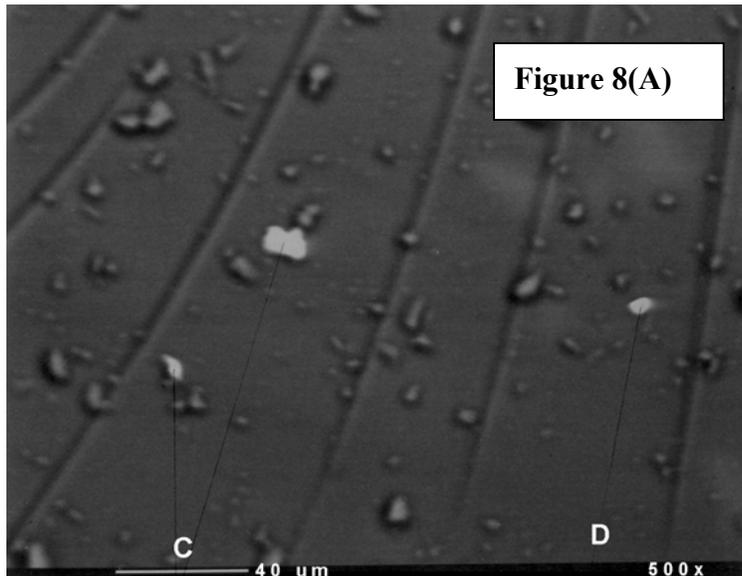


Table 13. Test #3 Rapid Cool Glass Composition

Env. B, AZ-102 Radioactive Glass									Average
Element	Acid % Elemental	Peroxide Fus., % Elemental	Oxide Oxide	Acid % % Oxide	Peroxide Fus. % Oxide	Target % Oxide	Acid % % Target	Peroxide Fus. % Target	% of Target
Al	3.2	3.3	Al ₂ O ₃	6.1	6.2	6.1	100	102	101
B	[1]	3.2	B ₂ O ₃	[1]	10.5	10.0	[1]	104	104
Ca	4.9	4.9	CaO	6.8	6.8	6.7	101	101	101
Cr	0.1	0.1	Cr ₂ O ₃	0.1	0.1	0.1			
Fe	4.3	4.3	Fe ₂ O ₃	6.1	6.1	5.3	115	114	115
K*	Not measured	Not measured	K ₂ O	0.2	0.2	0.2			
Li	2.8	2.8	Li ₂ O	6.1	6.0	5.9	104	103	103
Mg	1.9	1.9	MgO	3.1	3.2	3.0	104	106	105
Na	3.3	[2]	Na ₂ O	4.5	[2]	5.0	90	[2]	90
Si	26.3	23.2	SiO ₂	56.3	49.7	48.9	115	102	108
Ti	0.9	1.0	TiO ₂	1.6	1.7	1.4	110	123	116
Zn	2.6	2.8	ZnO	3.2	3.5	3.2	100	110	105
Zr	2.6	2.2	ZrO ₂	3.4	2.9	3.2	108	92	100
Cl*	Not measured	Not measured	Cl	0.0	0.0	0.0			
F*	Not measured	Not measured	F	0.1	0.1	0.1			
S*	Not measured	Not measured	SO ₃	0.8	0.8	0.8			
Totals:*				108.9	102.3	100.0			

Notes:

[1] Boron not measured in acid digestion due to use of boric acid

[2] Sodium not measured in sodium peroxide fusion due to use of sodium peroxide

* These elements not measured in dissolved glasses – target values assumed in sum of measured oxides

After the analyses of a limited portion of Test #3 glass were completed, the remaining amount of Test #3 glass was remelted and allowed to centerline cool using the same ~ 56 hour cooling profile that was used in previous Tests #1 and #2. The purpose of this Test #4 was to see if the bottom-forming crystalline surface could be formed on the previously rapid cooled glass if the longer ~ 56 hour cooling was applied. Photographs of the resulting remelted and canister cooled Test #4 glass are shown in Figure 9. These photographs show the glass to be dark and shiny on the top surface, with some visible crystalline material present on the bottom surface. The crystalline bottom surface material in this remelted and canister-cooled glass was less apparent than in the original two tests, Tests #1 and #2. Figure 10 presents the XRD analysis of the bottom surface of the Test #4 glass. The crystalline phase is best matched with crystal pattern number 80-1864 augite (See Table 12).

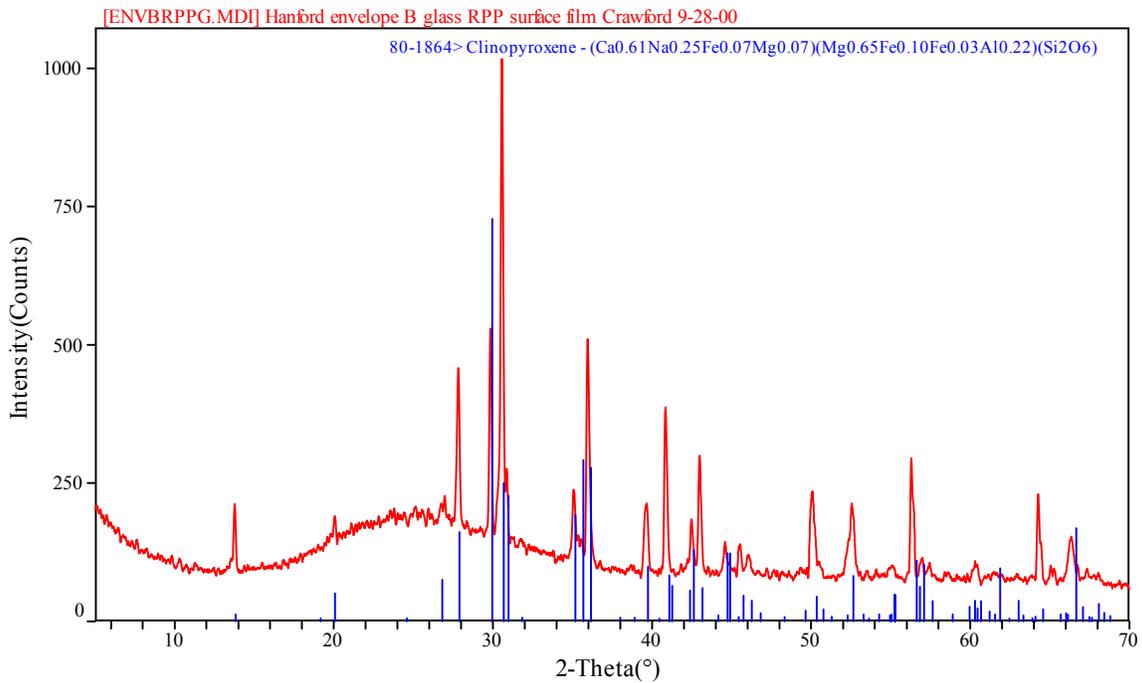
Figure 9. Photographs of Test #4 Glass (A) Top Surface and (B) Bottom Surface (A) Top Surface Test #4 Glass that was Remelted and Canister-Cooled Showing Amorphous Top Glass Surface.



(B) Bottom Surface Test #4 Glass that was Remelted and Canister-Cooled Showing Visible Crystalline Surface.



Figure 10. Test #4 Glass XRD Pattern of Bottom Surface Indicating Presence of Pyroxene Crystal Structure



Upon completion of Tests #1 - #4 and discussions with Hanford RPP and VSL personnel, it was decided to pursue two different tasks. The first task was for SRTC to prepare pieces of this Test #2 glass for XRD and microscopy analyses both at SRTC and at VSL. The purpose of this first task was to investigate the crystalline material on the bottom of the glass surface. The next task was for VSL to send samples of their AZ-102 simulants to SRTC for vitrification testing at SRTC. The purpose of this second task was to investigate the effects of crucible vitrifications carried out with VSL-supplied AZ-102 simulants in the sealed-quartz Deltech furnace at SRTC. These two tasks are discussed below.

First Task: Characterization of Radioactive Test #2 Glass by XRD and SEM/EDAX

Small shards of the Test #2 glass were prepared for XRD analysis and SEM/EDAX analysis. A 5-gram piece of the Test #2 glass was sent to VSL for analyses. VSL has recently completed their analyses of the glass that was sent from SRTC to VSL for testing. The results of the VSL analyses are currently being prepared in a separate report (VSL-01R4700-1, Rev. 0). All results presented and discussed below are from SRTC analyses of the Test #2 glass. A picture of the small glass pieces analyzed at SRTC is shown in Figure 11. The small glass pieces are numbered from Left to Right, as Samples #1, #2, #3 and #4, respectively. Figure 12 shows the XRD analysis of the bottom surface of the Test #2 glass. The bottom surface XRD shows the crystalline phase to be matched with an unnamed clinopyroxene with crystal structure pattern number 80-1862 (See Table 12).

Figure 11. Photograph of the Test #2 Radioactive AZ-102 Glass Fragments for XRD and SEM Analyses at SRTC (Dark Amorphous Glass with Yellow-Colored Crystal Surface on Each Piece)

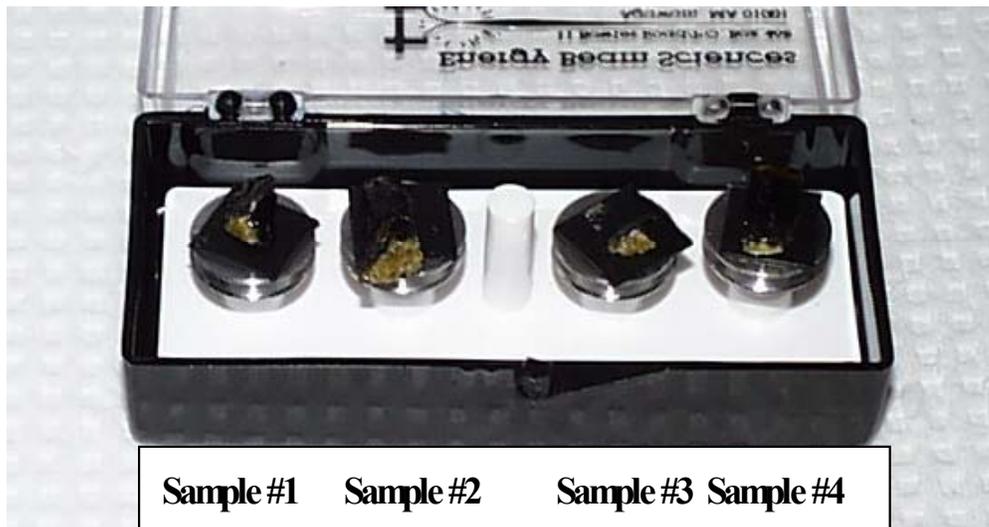
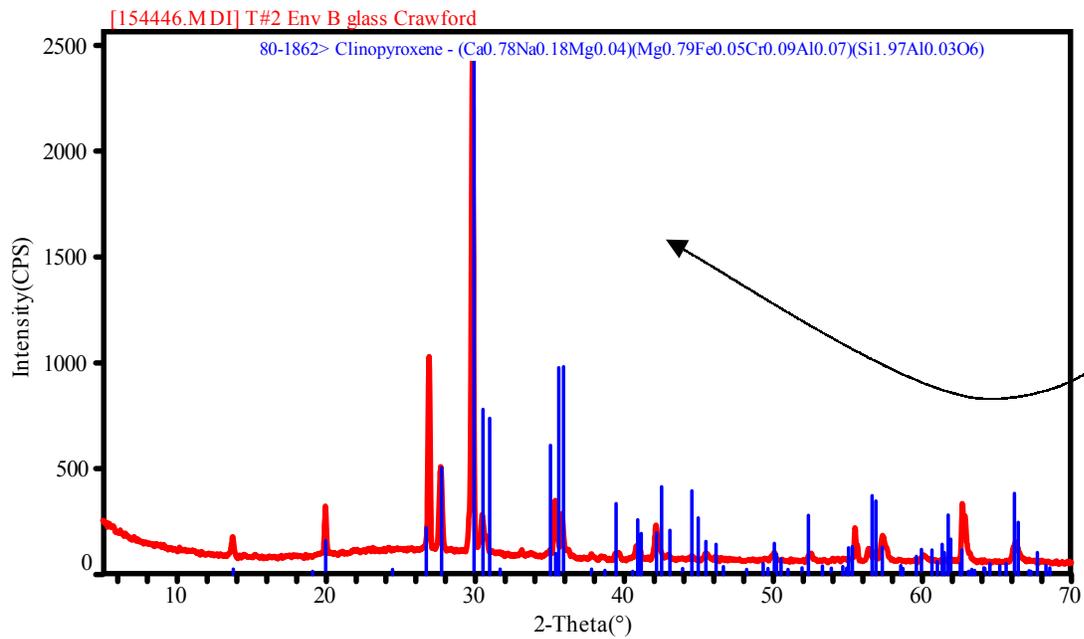


Figure 12. Test #2 Radioactive Glass XRD Pattern (SRTC Analysis) of Bottom-Forming, Yellow Crystalline Surface



SEM/EDAX data from Test #2 glass analyzed at SRTC are shown in Figures 13 and 14. The glass pieces shown earlier in Figure 11 were examined. Data from glass sample pieces 'Sample #2' are presented in Figure 13 and 'Sample #3' are presented in Figure 14. The following text gives explanation of each SEM or EDAX image shown in Figures 13 and 14.

Glass Sample #2 from Figure 11:

Figure 13 (A): 10X magnified SEM Secondary Electron (SE) image of Glass Sample #2.

Figure 13 (B): 10X magnified SEM Backscatter Electron (BSE) image of Glass Sample #2. Note the labeling of the crystalline region and the amorphous glassy region.

Figure 13 (C): 50X magnified SEM Backscatter Electron (BSE) image of Glass Sample #2. Spot A is the crystalline surface, Spot B is also the crystalline surface and Spots C are the very small light-colored specs in the image.

Figure 13 (D): EDAX of spot A from Figure 13 (C).

Figure 13 (E): EDAX of spot B from Figure 13 (C).

Note from the EDAX spectra in Figures 13(D) and 13(E), that the elements identified in this crystalline region are similar to the elements identified in the clinopyroxene phase from XRD (Ca, Mg, Fe, Cr, and Si) as well as the major elements in the glass (from Table 13, Ca, Mg, Fe, Al and Si).

Figure 13 (F): EDAX of spot C from Figure 13 (C).

Note that the EDAX spectra in Figure 13(F) indicates presence of platinum associated with the very small light spots shown as 'Spots C' in Figure 13(C).

Glass Sample #3 from Figure 11:

Figure 14 (A): 10X magnified SEM Backscatter Electron (BSE) image of Glass Sample #3.

Figure 14 (B): 10X magnified SEM Secondary Electron (SE) image of Glass Sample #3.

Figure 14 (C): 25X magnified SEM Backscatter Electron (BSE) image of Glass Sample #3.

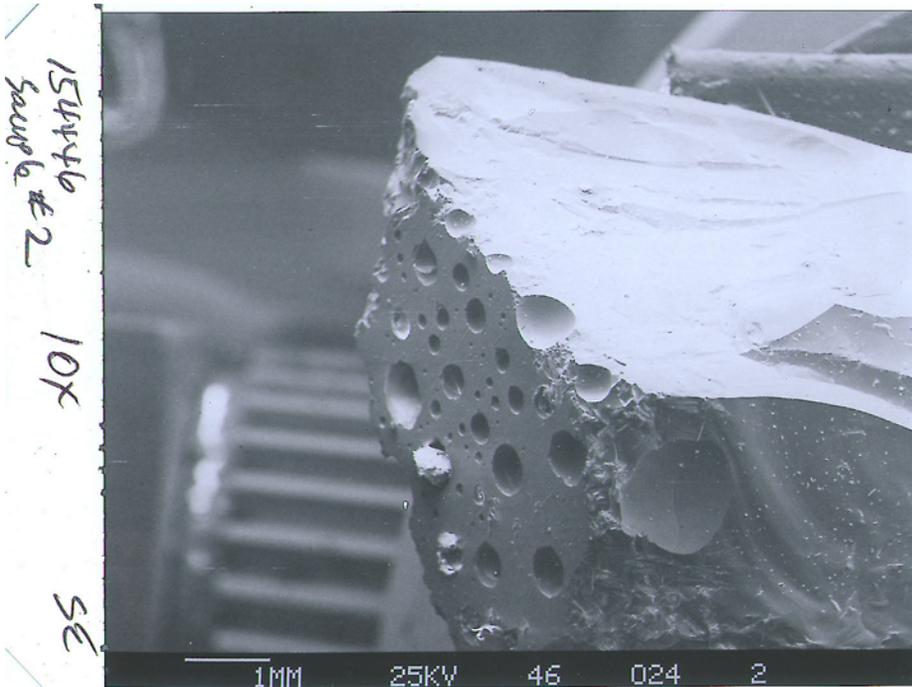
Figure 14 (D): 25X magnified SEM Secondary Electron (SE) image of Glass Sample #3.

Figure 14 (E): EDAX of spot A from Figure 14 (C).

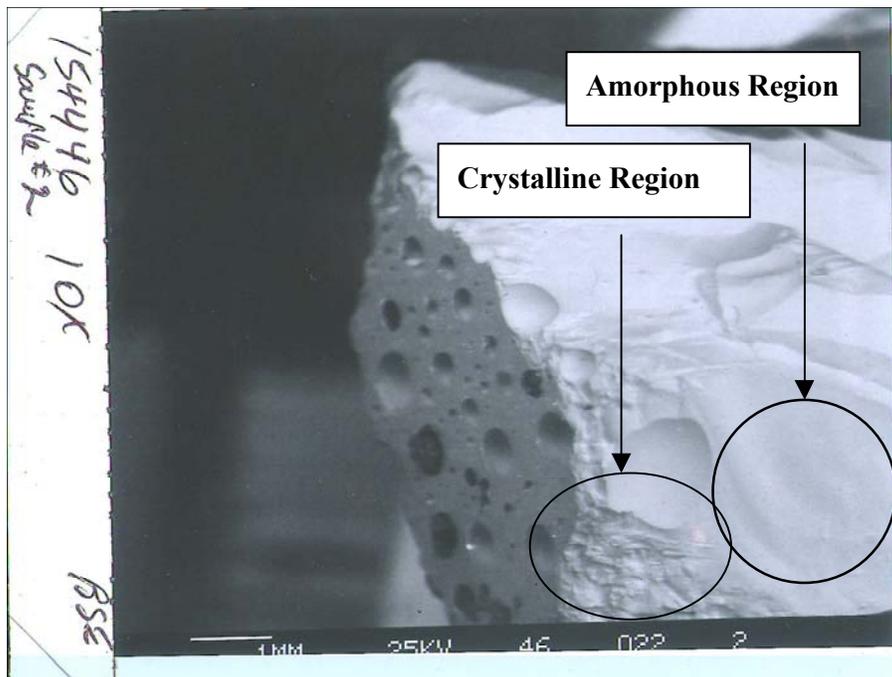
Figure 14 (F): EDAX of spot B from Figure 14 (C).

Figure 13. SEM and EDAX Photos (SRTC Analysis) of Test #2 Glass, Sample #2 from Figure 11

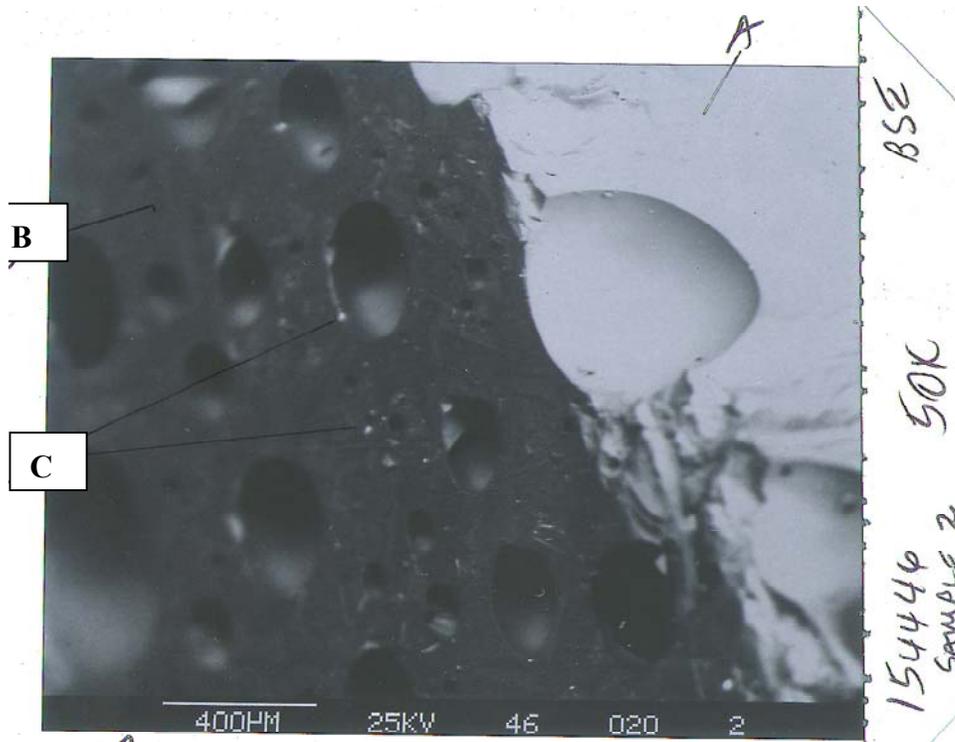
(A) SEM Secondary Electron (SE) 10X Magnified Image



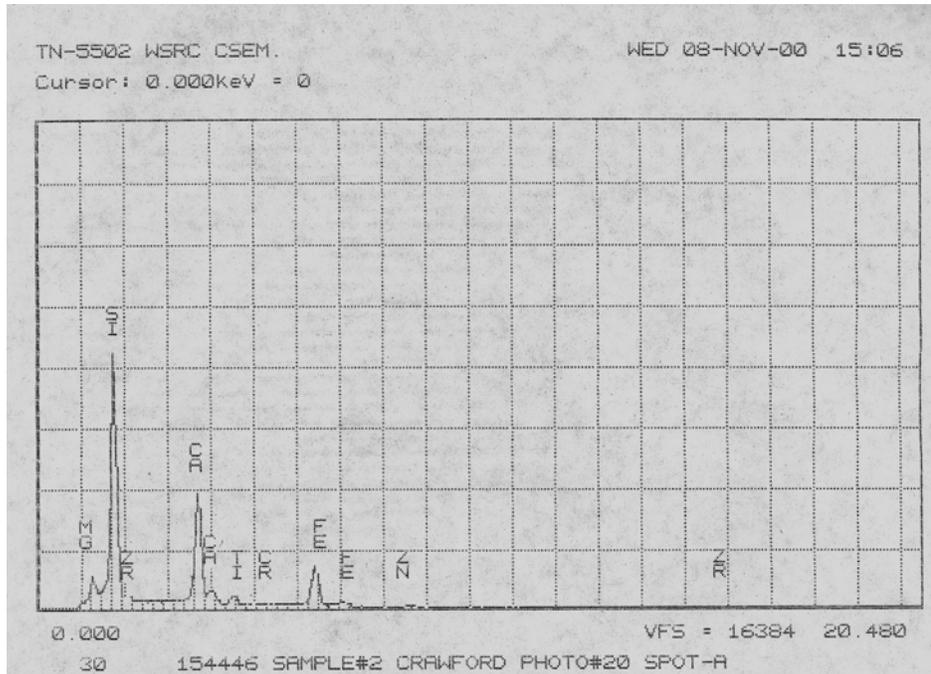
(B) SEM Backscatter Electron (BSE) 10X Magnified Image



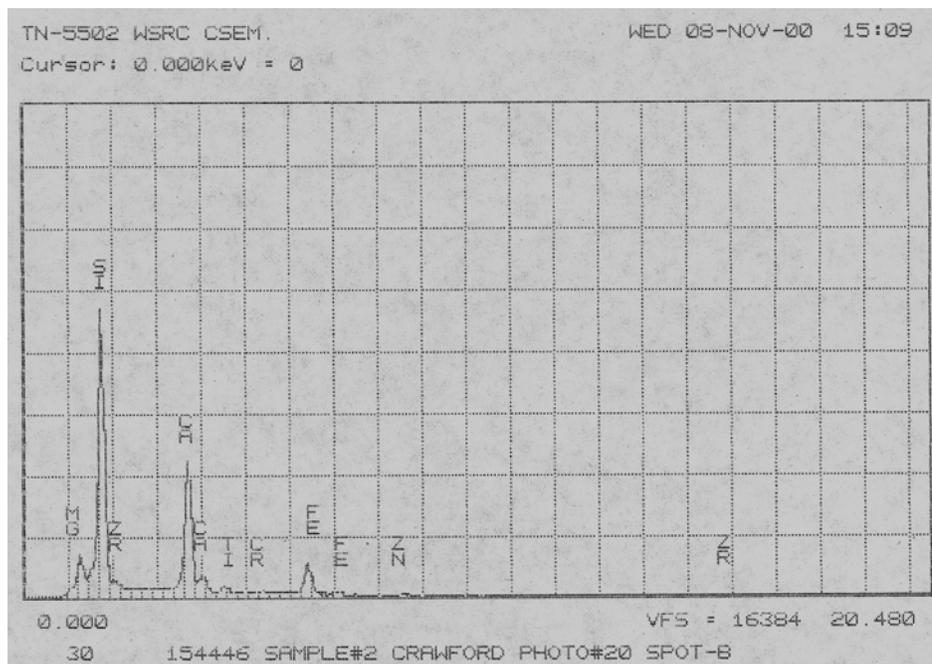
(C) SEM Backscatter Electron (BSE) 50X Magnified Image. Spot A is the crystalline surface, Spot B is also the crystalline surface and Spots C are the very small light-colored specs in the image.



(D) EDAX Spectrum from Spot A in Figure 13-C.



(E) EDAX Spectrum from Spot B in Figure 13-C



(F) EDAX Spectrum from Spots C in Figure 13-C

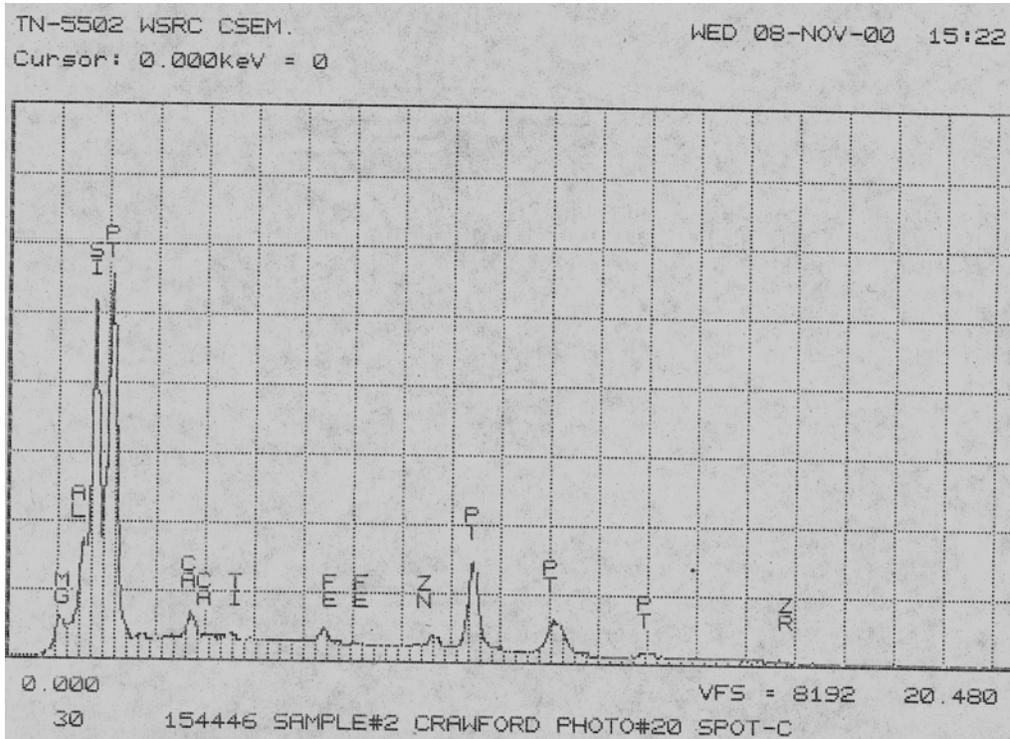
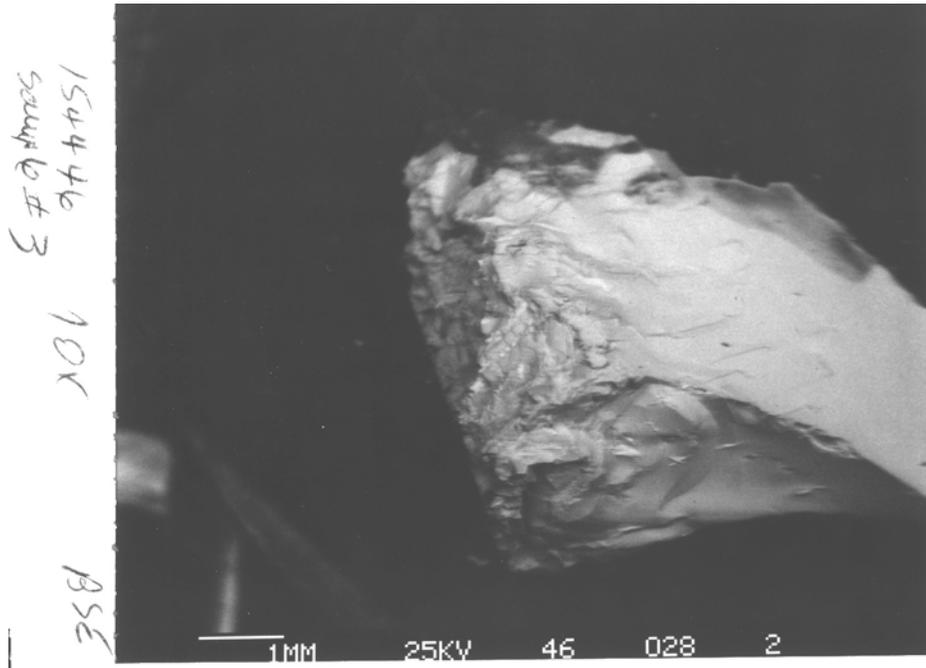
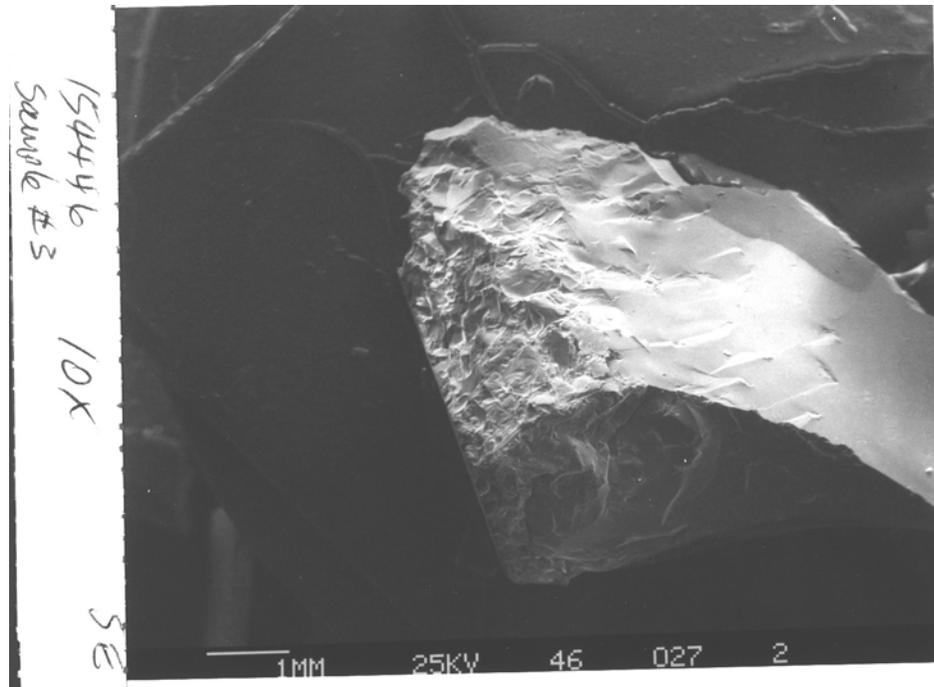


Figure 14. SEM and EDAX Photos (SRTC Analyses) of Test #2 Glass, Sample #3 from Figure 11

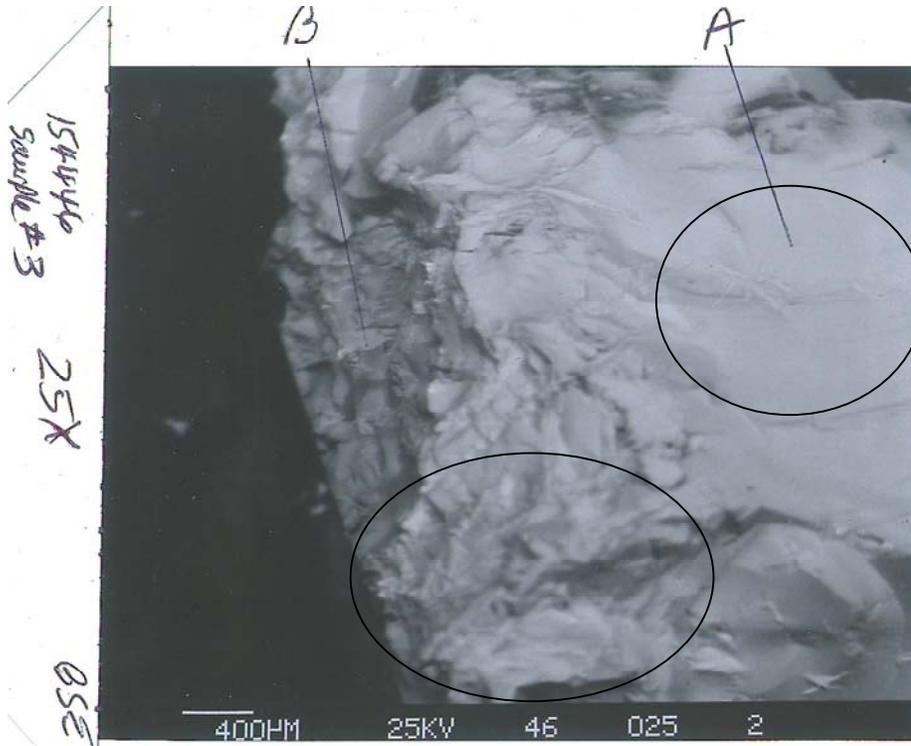
(A) SEM Backscatter Electron (SE) Image



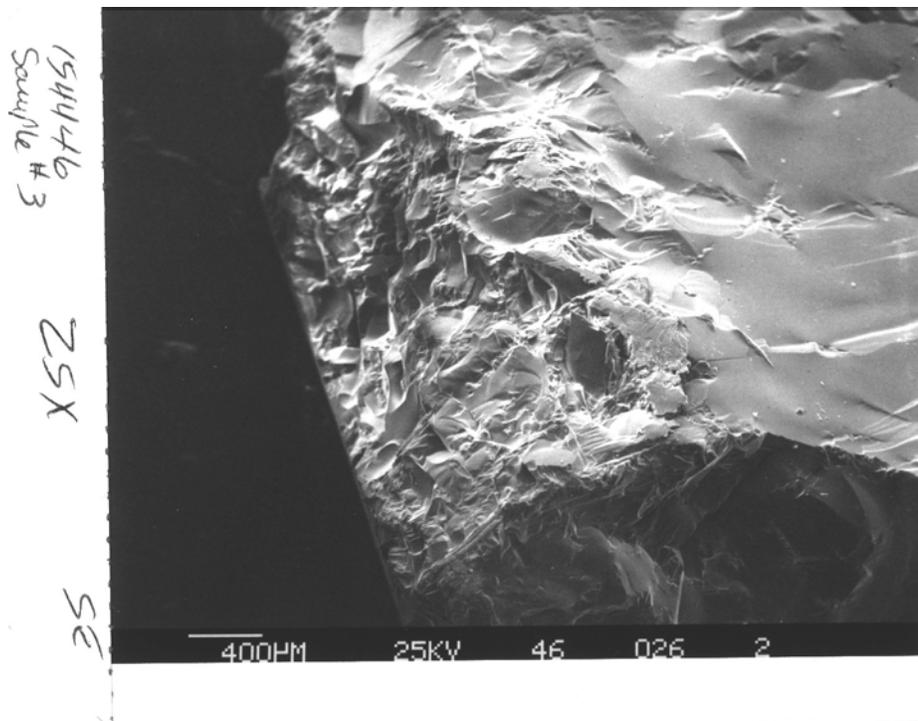
(B) SEM Secondary Electron (SE) Image



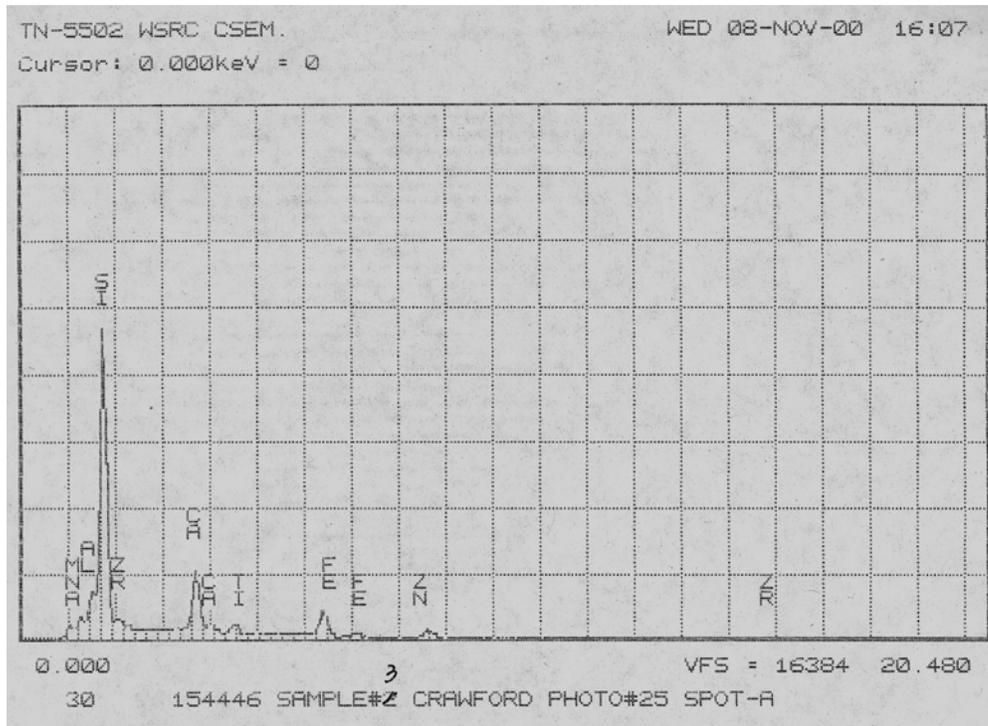
(C) SEM Backscatter Electron (SE) Image. Region marked 'A' is amorphous glassy region and region marked 'B' is crystalline layer region.



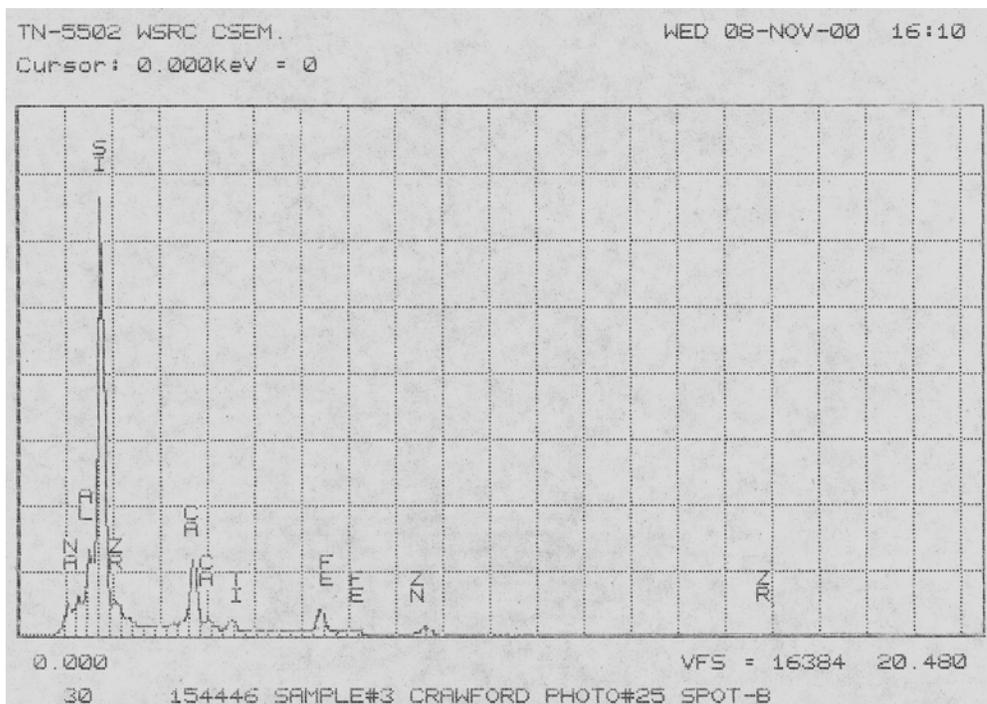
(D) SEM Secondary Electron (SE) Image



(E) EDAX Spectra from Spot A in Figure 14 (C).



(F) EDAX Spectra from Spot B in Figure 14 (C).



One can estimate the total volume percent of the pyroxene crystalline phase in the Test #2 glass using the SEM data shown in Figures 13 and 14. The thickness of the crystalline phase is estimated to be on the order of between 0.5 mm and 1 mm (See Figure 13(B) and Figure 14(C)). For calculation purposes, one can use an average of about 0.075 cm. Other glass dimensions are top diameter = 7.5 cm, bottom diameter = 6 cm and thickness of overall glass puck = 1.2 cm. If one conservatively assumes that the crystalline bottom-forming layer is uniform across the entire bottom of the glass puck, then Equation 2 below can be used to estimate the vol% of the total puck that is occupied by the bottom-forming crystalline layer. Since the observed bottom crystalline layer is not distributed entirely across the bottom of the glass pucks (See Figures 4(B) and 4(C)), then the calculated 5 vol% crystalline content of the glass is estimated to be an upper limit.

Equation 2:

$$\text{Vol\% crystalline} = (\text{volume of crystalline layer} / \text{total glass puck volume}) * 100$$

$$\begin{aligned} \text{With: volume of crystalline layer} &= \pi * r^2 * \text{height} \\ &= 3.14 * (6\text{cm}/2)^2 * 0.075 \text{ cm} \\ &= 2.12 \text{ cm}^3 \end{aligned}$$

$$\begin{aligned} \text{volume of total glass puck} &= \pi * r^2 * \text{height} \\ &= 3.14 * (6.75\text{cm}/2)^2 * 1.2 \text{ cm} \\ &= 42.92 \text{ cm}^3 \end{aligned}$$

$$\text{Vol\% crystalline} = (2.12 \text{ cm}^3 / 42.92 \text{ cm}^3) * 100 \approx 5 \text{ vol\%}$$

Second Task: Vitrification of VSL Simulants at SRTC, Tests #5 and #6

Table 14 shows the glass formulation for the surrogate material received from VSL for crucible testing at SRTC. The data shown in Table 14 is reproduced from the spreadsheet information that accompanied the two surrogate samples delivered to SRTC from VSL. The material was received in two forms, a dry powder blended mixture (LAWB53SUR2) and a glass (LAWB53SUR1). The LAWB53SUR1 glass sample had been vitrified (platinum/gold crucible, no stirring, quench cooled) at VSL before shipment to SRTC. Each material was heated, melted and centerline cooled in the SRTC Deltech sealed-quartz crucible furnace described above. The dry powder mix LAWB53SUR2 (Test #5) was heated, melted and cooled in similar fashion as preceding Tests #1 - #3. The VSL simulant glass sample LAWB53SUR1 (Test #6) was heated, melted and cooled similar to the Test #4 previous glass remelt test. The resulting glasses produced in the VSL simulant testing are shown in Figures 15 - 16. These glasses had dark and shiny top surfaces and there was minimal visible bottom-surface crystalline material present. The resulting glass monoliths were analyzed by XRD for presence of crystalline structure on the top and bottom surfaces. Results shown in Figures 17-18 are from XRD analyses of the vitrified LAWB53SUR2 powder sample (Test #5). Figures 19-20 are from XRD

analyses of the vitrified LAWB53SUR1 glass sample (Test #6). XRD analysis of the bottom surfaces of glasses from both Test#5 (Figure 18) and Test#6 (Figure 20) showed the crystalline phase to be matched with crystal pattern # 80-1864 augite (See Table 12). It should be noted that previous melter testing with a mixed tank AZ-101/AZ-102 surrogate blends have also produced glasses with the augite secondary phase, in addition to other phases reported (zircon, chromite, spinel, apatite and rutile) [13]. One should use caution in comparing the present AZ-102 radioactive crucible-scale vitrification study secondary phase formation to previous melter study glasses with blended AZ-101/AZ-102 surrogates. Numerous differences exist between the two studies. The melter work at VSL used surrogates with a blended tank best basis inventory (vs. radioactive AZ-102 crucible work at SRTC) and the melter studies were targeted at investigating the extremes of sulfate loading in the glasses.

Table 14. VSL Surrogate Material for Vitrification Tests #5 and #6 at SRTC.

Recipe for VSL Surrogate				
Component		Amount (g)	Glass Oxide	Loading
Al ₂ O ₃		6.1	Al ₂ O ₃	6.0987
H ₃ BO ₃		17.99	B ₂ O ₃	10.0256
CaCO ₃		12.16	CaO	6.7461
Cr ₂ O ₃		0.11	Cr ₂ O ₃	0.1135
Fe ₂ O ₃		5.535	Fe ₂ O ₃	5.3406
K ₂ CO ₃		0.34	K ₂ O	0.2269
Li ₂ CO ₃		14.79	Li ₂ O	5.8559
MgO		3.12	MgO	2.9982
Na ₂ CO ₃		7.3	Na ₂ O	5
SiO ₂		47.61	SiO ₂	48.9243
TiO ₂		1.42	TiO ₂	1.4054
ZnO		3.2	ZnO	3.1857
ZrSiO ₄		5.07	ZrO ₂	3.1856
NaCl		0.01	Cl	0.0049
NaF		0.14	F	0.0633
Na ₂ HPO ₄		0.03	P ₂ O ₅	0.0134
Na ₂ SO ₄		1.46	SO ₃	0.8072
		SUM		99.9953

Figure 15. Photographs of Vitrified Product Glass from Dry Powder VSL Simulant (Test #5)

Top image shows dark amorphous top glass surface with no visible crystals. Bottom image shows visible crystals on bottom of glass piece.

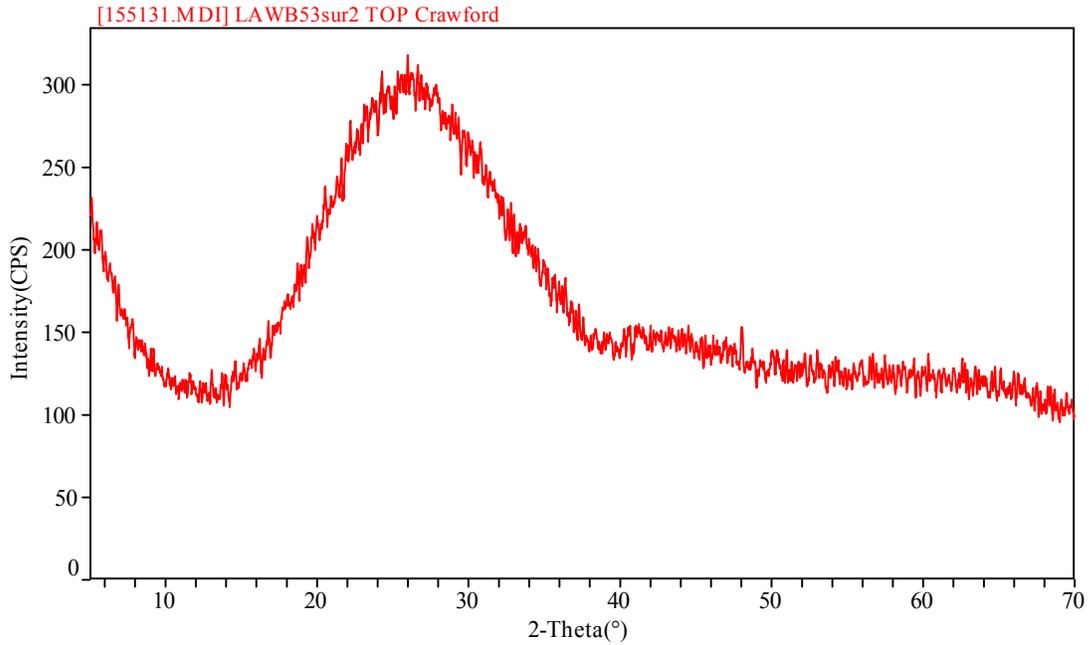


Figure 16. Photographs of Vitrified Product Glass from Remelting of VSL Simulant Glass (Test #6)

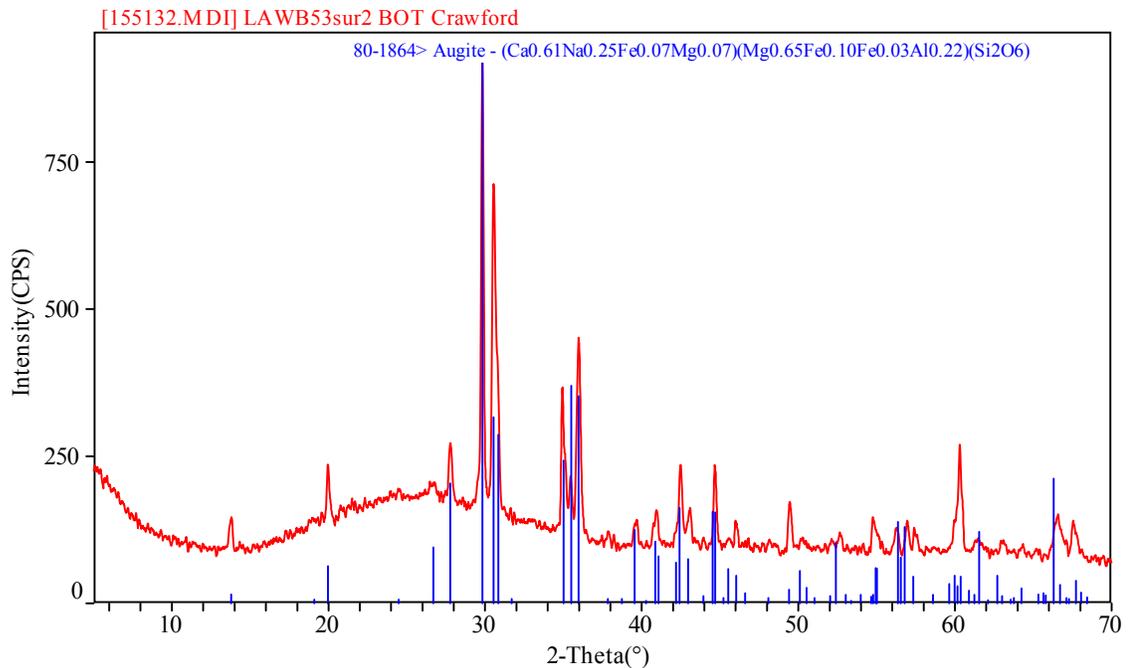
Visual observation of the top image (top glass surface) and bottom image (bottom surface) indicated no significant presence of crystals.



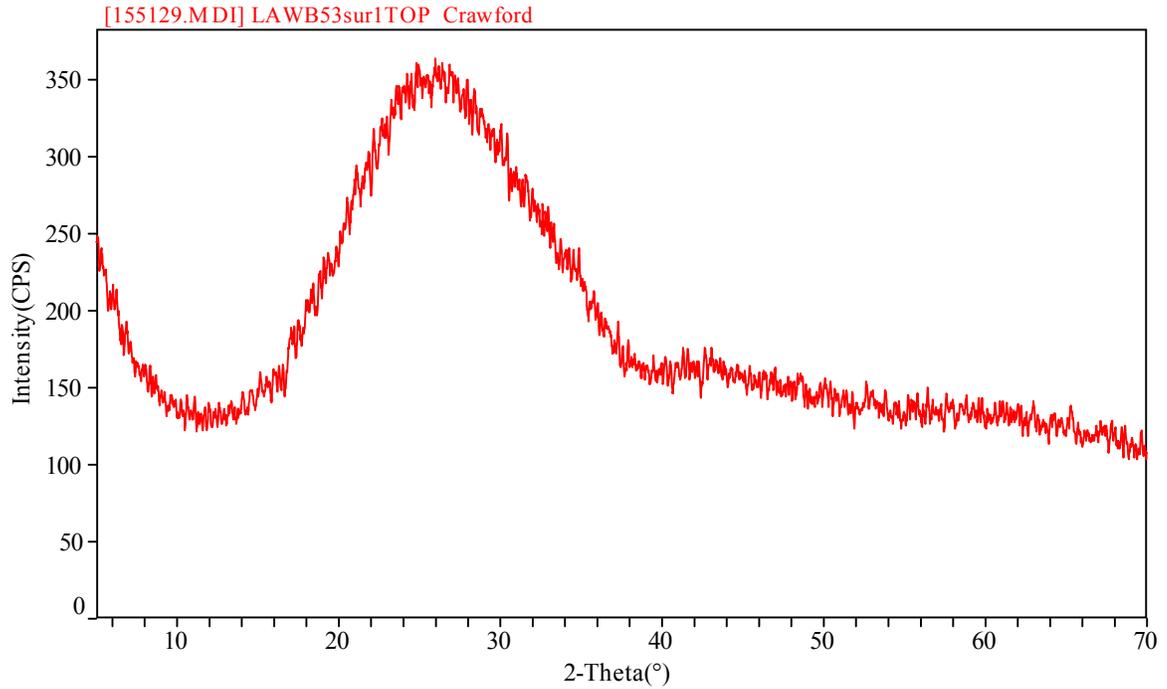
**Figure 17. XRD Spectra of Top of Vitrified Glass Made from VSL Glass
LAWB53SUR2 in Test #5
(See Figure 15 top image)**



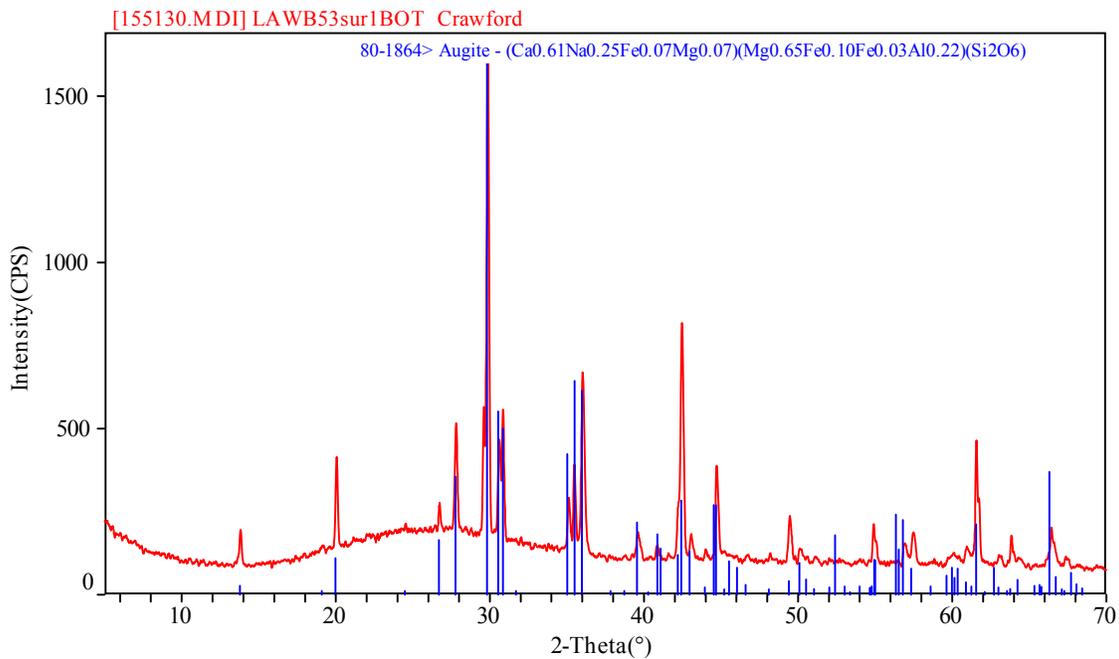
**Figure 18. XRD Spectra of Bottom of Vitrified Glass Made from VSL Glass
LAWB53SUR2 in Test #5
(See Figure 15 bottom image)**



**Figure 19. XRD Spectra of Top of Vitrified Glass Made from VSL Glass
LAWB53SUR1 in Test #6
(See Figure 16 top image)**



**Figure 20. XRD Spectra of Bottom of Vitrified Glass Made from VSL Glass
LAWB53SUR1 in Test #6
(See Figure 16 bottom image)**



Vitrification Testing with SRTC-Developed AZ-102 Simulant, Tests #7 - #9

Data described above for vitrification Tests #1 - #6 were considered for review by Hanford RPP, SRTC and VSL personnel. Although crucible melts of the VSL AZ-102 simulants produced glasses that contained bottom-forming crystalline material as identified by XRD techniques, these crystals did not appear to be present to the same extent as was seen on the initial radioactive melts in Tests #1 and #2. In other words, the VSL AZ-102 simulant qualitatively reproduced the crystalline phase observed in radioactive testing, but it did not appear to quantitatively reproduce the amount of observable secondary crystalline phase present on the bottom of the glass melts. It was decided to pursue development of a more representative simulant material for further vitrification testing. The goals for further research and testing were to first develop a simulant that could both qualitatively and quantitatively reproduce the observed bottom-forming crystalline phase seen in radioactive tests. Assuming this could be accomplished, the next goal was to use the newly developed simulant in a modified furnace configuration.

The original radioactive AZ-102 concentrated supernate (Table 5) was submitted for further analyses at SRTC. These analyses consisted of various organic chromatography techniques aimed at characterization of the dissolved carbon species in the waste. Total organic and total inorganic carbon were also reanalyzed. The resulting data are shown in Table 15 in the column labeled Target Concentration AZ-102 Radioactive. Total organic carbon values for the AZ-102 sample ranged from 19 g/L +/- 7 g/L for the initial analyses (Table 5) and 10 g/L for the second analyses (Table 15). Chromatographic techniques indicated that the total organic carbon in AZ-102 is comprised of acetate, citrate, formate, glycolate and EDTA.

Characterization data shown in preceding Table 5 and the latter characterization data pertaining to the specific organics shown in Table 15 were transmitted to Russ Eibling of SRTC to develop a simulant solution for the AZ-102 sample. Spreadsheets pertaining to the revised AZ-102 simulant are presented in Appendix D. Previous work with Env. B simulants at SRTC produced an AZ-102 simulant solution that had only oxalate as added organic source. The added oxalate was only 13% of the total organic carbon measured in actual AZ-102 radioactive samples [6,14-15]. The revised AZ-102 simulant was prepared and characterized and the data is shown in column 'Measured Concentration AZ-102 Simulant' Table 15 compared to the radioactive AZ-102 data. Comparison of the data for radioactive AZ-102 with the simulant data shown in Table 15 indicates good match between most analytes in the two systems. The analytes aluminum and oxalate were very low in the revised simulant relative to the measured values in the radioactive sample. The analytes fluoride and citrate were very high in the revised simulant analysis relative to the measured values in the radioactive sample. It was not further investigated why these particular elements were so extremely off target. One possible explanation for the low aluminum in the simulant vs. target is the use of hydrated aluminum trihydroxide powder that was formulated as the anhydrous $Al(OH)_3$ powder. More recent simulant

preparations use aluminum nitrate nonahydrate ($\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$). Since the majority of the analytes shown in Table 15 including the sodium, anions and the organic complexants were close to target, it was decided to proceed with this revised simulant for use in further vitrification tests. It should be noted that current RPP/WTP plans for all current and future surrogate work involve development of RPP/WTP approved simulants that can be used throughout the research and testing studies at SRTC, Battelle and VSL/CUA. Thus future work with simulants of Tank AZ-102 or any other tanks will likely involve better matching to the target analytes for all species in the simulant.

Photographs of the radioactive AZ-102 supernate and the revised simulant AZ-102 supernate are shown in Figure 21. Initial attempts to formulate the AZ-102 simulate produced a green colored liquid. However, further attempts with different order of addition for chemicals produced the appropriate yellow colored solution shown in Figure 21. The difference in the original green colored supernate and the final yellow colored supernate are most likely related to the oxidation state of dissolved chromium in solution.

Table 15. Comparison of Significant Components in Radioactive AZ-102 Supernate with Simulant AZ-102 Supernate

Component	Target* Concentration AZ-102 Radioactive		Measured** Concentration AZ-102 Simulant		% of Target
	(mg/L)		(mg/L)		
Aluminum	798		97		12.2
Calcium	67		53.3		79.6
Carbonate	46944		45503		96.9
Oxalate	4625		1984		42.9
Cesium	0.1				
Chromium	1018		1020		100.2
Fluoride	1370		2808		205.0
Magnesium	3.1		<0.8		
Molybdenum	79		84		106.3
Nitrate	27305		26969		98.8
Nitrite	49440		48173		97.4
Potassium	4555		4412		96.9
Silicon	56.0		64		114.3
Sodium	100820		102330		101.5
Strontium	0.1				
Sulfate	26300		25097		95.4
TIC	9396		10900		116.0
TOC***	5000-25000		10030		
Acetate	1050		1041		99.1
Citrate	6803		12000		176.4
Formate	7270		7537		103.7
Glycolate	13560		12590		92.8
HEDTA	<250				
EDTA	350		544		155.4

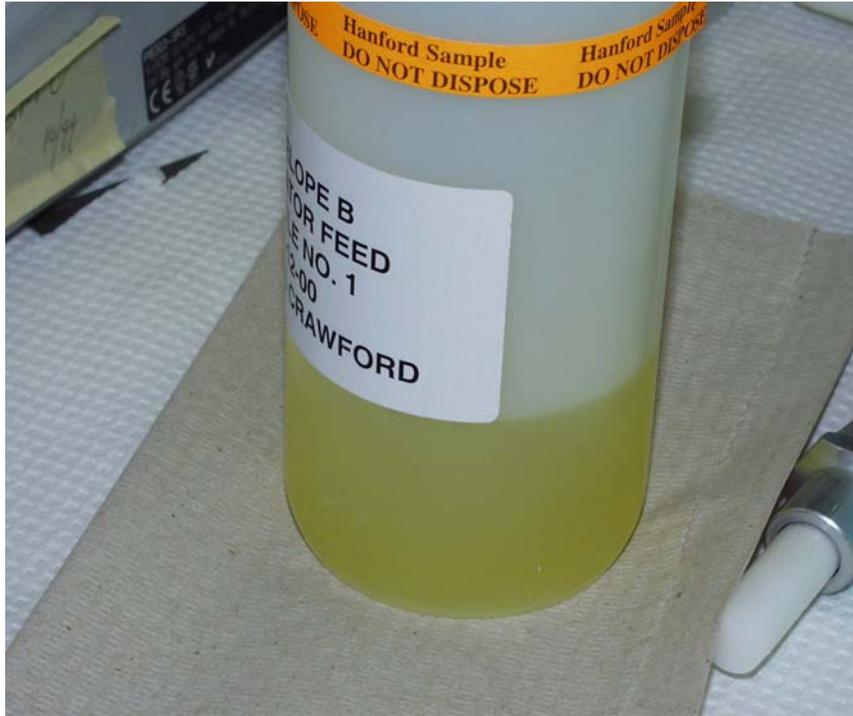
* Target concentration values from Table 5 and Appendix D

** Measured concentrations from analysis of AZ-102 simulant

*** TOC values for radioactive AZ-102 ranged from 5 – 25 g/L. Measured value in AZ-102 simulant = 10 g/L compared to calculated value of 12 g/L.

Figure 21. Photographs of Radioactive AZ-102 Supernate and the Revised Simulant AZ-102 Supernate

(A) Radioactive AZ-102 Supernate



(B) Revised Simulant AZ-102 Supernate



Two vitrification tests were next performed with the new revised AZ-102 simulant. Tests #7 and #8 used the revised AZ-102 simulant at the same waste loading that was used in previous radioactive Tests #1 - #3. Test #7 was carried out in the Deltech sealed quartz furnace with Pt/Au crucible and Test #8 was carried out in a standard benchtop Thermolyne furnace with Pt/Au crucible. The purpose of performing Test #7 was to compare the behavior of the revised AZ-102 simulant with previous radioactive Tests #1 - #2. The purpose of performing Test #8 in parallel with Test #7 was to compare the behavior of the revised simulant in a sealed-quartz configuration (Test #7) vs. an 'open' atmosphere inside a standard furnace on the benchtop (Test #8).

Each crucible melt in Test #7 and #8 was performed with identical heatup, melt and simulated canister cooling. Resulting glasses were examined for visible secondary crystalline phase production. Both of these glasses contained dark shiny top surfaces and visible yellow-colored bottom-forming crystalline surface. Figures 22 and 23 show the bottom-forming crystalline phase similar to earlier observed yellow-colored bottom-forming crystalline phases on radioactive AZ-102 melts. Figure 22 shows the Test #7 simulant glass on the left vs. the Test #2 radioactive glass on the right. Figure 23 shows the Test #8 simulant glass. The XRD patterns from these two Test #7 and #8 glasses are shown in Figures 24 and 25. The XRD data confirms visual observation of the glasses in that the top surface XRD patterns (Figure 24A and 25A) show little to no crystalline phase and the bottom surface XRD patterns (Figure 24B and 25B) shows crystalline structure that is best matched with crystal pattern # 80-1864 augite (See Table 12).

Results from Tests #7 and #8 indicated that indeed the revised AZ-102 simulant behaved similarly to earlier vitrification testing with the actual radioactive AZ-102 supernate from Tests #1 and #2. Comparison of Test #7 and #8 glasses showed that the bottom-forming phase could be readily produced from either the Deltech sealed-quartz furnace configuration or the open crucible melt inside a standard benchtop Thermolyne furnace. There also appears to be less total amount of crystalline material in the Test #8 glass made in the open crucible vs. the Test #7 glass that was made in the sealed quartz Deltech furnace. These tests also show that the revised simulant was adequate for the purposes of this study despite the extremely large differences between targeted and analyzed analytes such as Al, oxalate, F, and citrate discussed above (Table 15).

The redox state of the bulk glass was measured for the Test #8 simulant glass. This analysis uses powdered glass that is dissolved and analyzed for Fe^{2+} and Fe^{3+} spectrophotometrically. A standard Environmental Assessment (EA) glass is run in parallel. Results of these tests on two different portions of the Test #8 glass piece are shown in Table 16. The ratio of Fe^{2+} to Fe^{3+} ($\text{Fe}^{2+}/\text{Fe}^{3+}$) for the Test #8 glass indicates a relatively low value in the range of ~ 0.035 . The standard EA glass $\text{Fe}^{2+}/\text{Fe}^{3+}$ ratio was measured to be very close to the reported average value of 0.22 ± 0.1 [16]. These data indicate that the bulk glass produced in Test #8 is very oxidized, even though the augite crystalline structure identified on the bottom glass surface is enriched in the reduced Fe^{2+} (See Table 12). Redox measurements have been reported in VSL melter studies with blended tank AZ-101/AZ-102 surrogate feeds [13]. Redox values ($\text{Fe}^{2+}/\text{Fe}(\text{total})$) for

glasses studied in that work range from 0.034 (more oxidized glass) to 0.442 (more reduced glass). One should use caution in comparing the present AZ-102 radioactive crucible-scale vitrification study to previous melter studies with blended AZ-101/AZ-102 surrogates. Numerous differences exist between the two studies. The melter work at VSL used surrogates with a blended tank best basis inventory (vs. radioactive AZ-102 crucible work at SRTC) and the melter studies were targeted at investigating the extremes of redox in the blended AZ-101/AZ-102 surrogate.

Table 16. Results of Redox Analysis on Test #8 Simulant Glass

Glass	Fe ²⁺	Fe(total)	Fe ²⁺ /Fe ³⁺	Fe ²⁺ /Fe(total)
Env. B Replicate # 1	0.015	0.404	0.038	0.036
Env. B Replicate # 2	0.012	0.378	0.032	0.031
EA	0.130	0.722	0.220	0.180

Figure 22. Comparison of the Bottom-Forming Crystalline Phase from Test #7 Simulant Glass (Left) and the Bottom-Forming Crystalline Phase from Test #2 Radioactive Glass (Right)

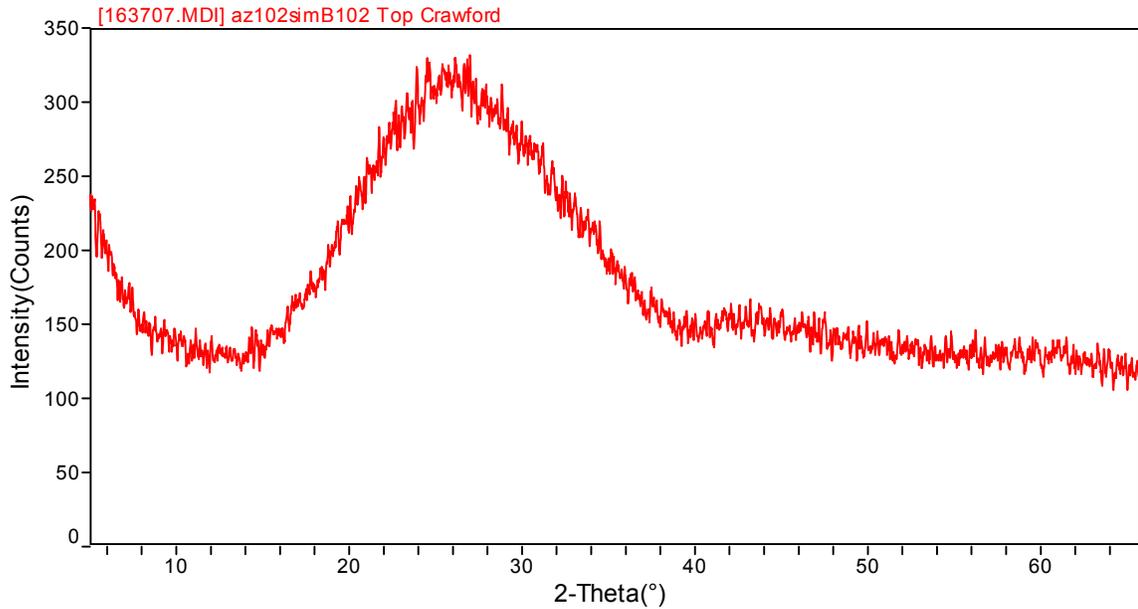
Each glass was produced in the sealed-quartz Deltech furnace.



Figure 23. The Bottom-Forming Crystalline Phase from Test #8 Simulant Glass Produced in a Thermolyne Furnace with Open Crucible



Figure 24. XRD Pattern of the Top and Bottom of Test #7 Glass Made in Sealed Quartz Deltech Furnace with Revised AZ-102 Simulant
(A) Top surface XRD pattern showing no crystalline peaks.



(B) Bottom surface XRD pattern showing presence of augite crystal.

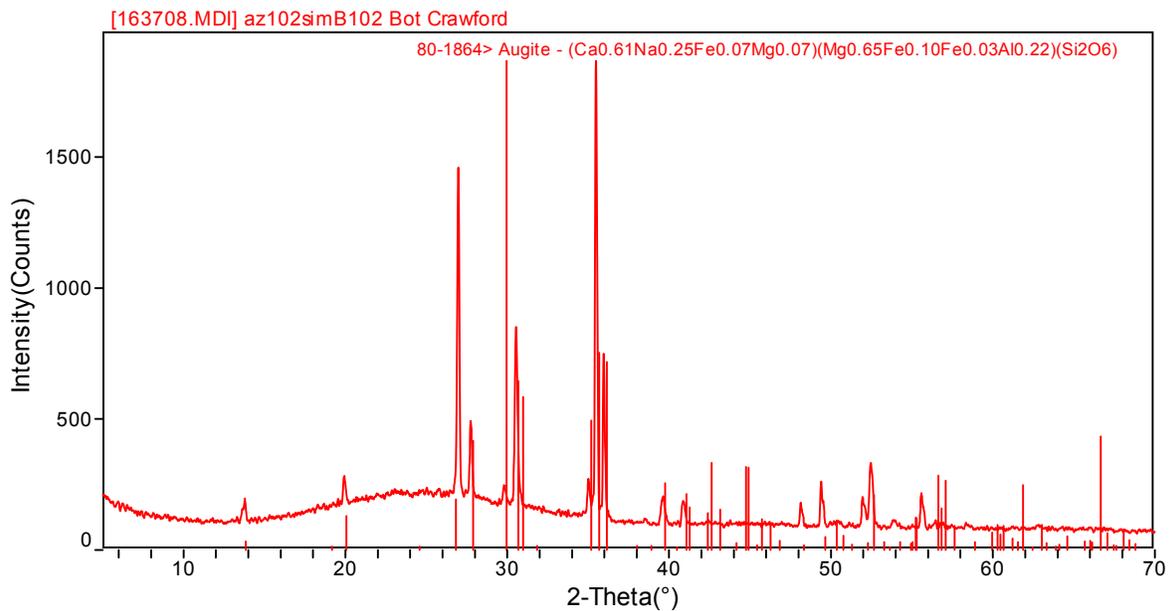
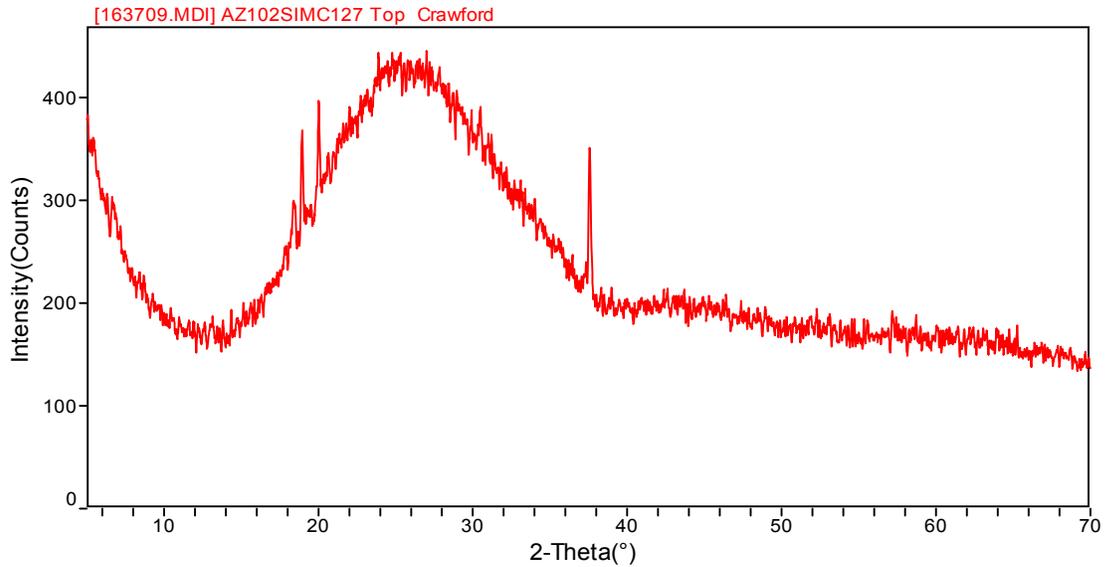
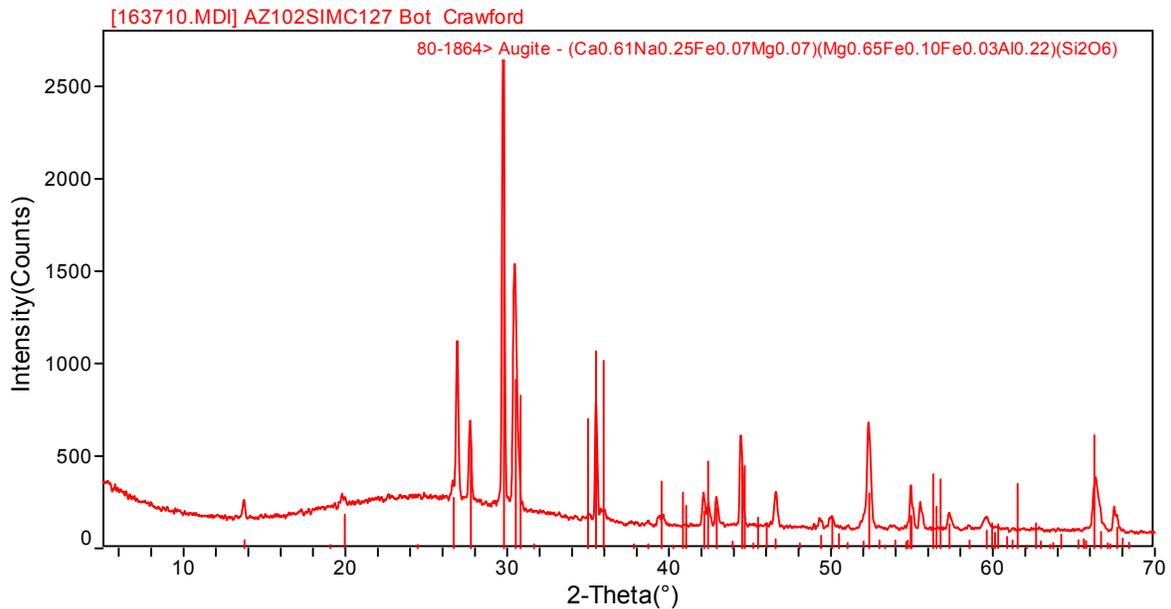


Figure 25. XRD Pattern of the Top and Bottom of Test #8 Glass Made in Open Crucible Inside of Thermolyne Furnace on Benchtop

(A) Top surface XRD pattern showing predominant amorphous structure. The 3 to 4 small peaks in XRD pattern were not identifiable by attempts to match this pattern with XRD-library spectrum data.



(B) Bottom surface XRD pattern showing presence of augite crystal.



A final vitrification Test #9 was performed with the revised AZ-102 simulant in a modified sealed-quartz furnace configuration using the Deltech furnace. Modifications to the setup included (1) a larger diameter air-inlet tube to allow more air into the system, (2) placement of the outlet port of the air-inlet tube closer to the crucible melt surface to allow for closer placement of incoming air to the actual glass melt surface, and (3) a silica gel drying column added to the air-inlet line to adsorb moisture in the incoming ambient air. This last modification was done to allow for a higher flux of dryer air through the system. Previous attempts at using increased airflow, i.e., higher vacuum levels in the offgas system, resulted in excess condensate forming in the offgas system. The excess offgas condensate was due to the condensation of humid ambient air since this effect was often noticed before crucible melt slurries had reached boiling temperatures.

The crucible melt in #9 was performed with identical heatup, melt and simulated canister cooling as was used in previous Tests #1 - #2 and Tests #7 - #8. The resulting glass was examined for visible secondary crystalline phase production. Figure 26 shows the top and bottom images of the glass. The top surface shown in Figure 26A indicates a dark and shiny surface. The bottom surface of the glass shown in Figure 26B contains visible crystalline surface areas – although to a much less extent than was observed in earlier Tests #1 - #2 and Tests #7 - #8. There appears to be some yellow-colored crystalline material in the upper right quadrant of the bottom surface of the glass piece shown in Figure 26B. The top and bottom surface of this Test #9 glass was examined by XRD and the results are shown in Figure 27A for the top surface and Figure 27B for the bottom surface. The augite crystalline phase pattern # 80-1864 was identified by XRD (See Table 12).

Figure 26. Test #9 Glass Vitrified with AZ-102 Simulant in Modified Deltech Sealed Quartz Furnace

Figure 26(A): Top View of Glass Puck Showing Dark Shiny Amorphous Surface

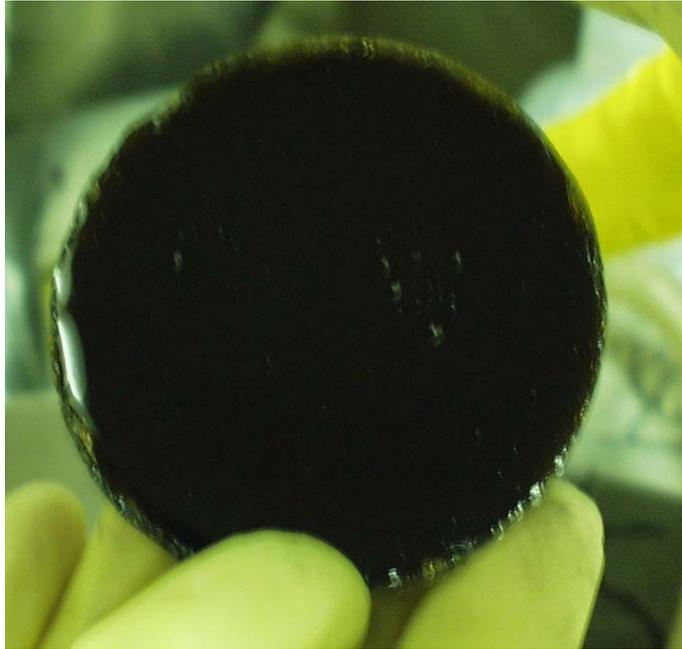


Figure 26(B): Bottom View of Glass Puck Surface Showing Slight Visible Crystalline Material in Upper Right Quadrant of Glass Puck Surface.

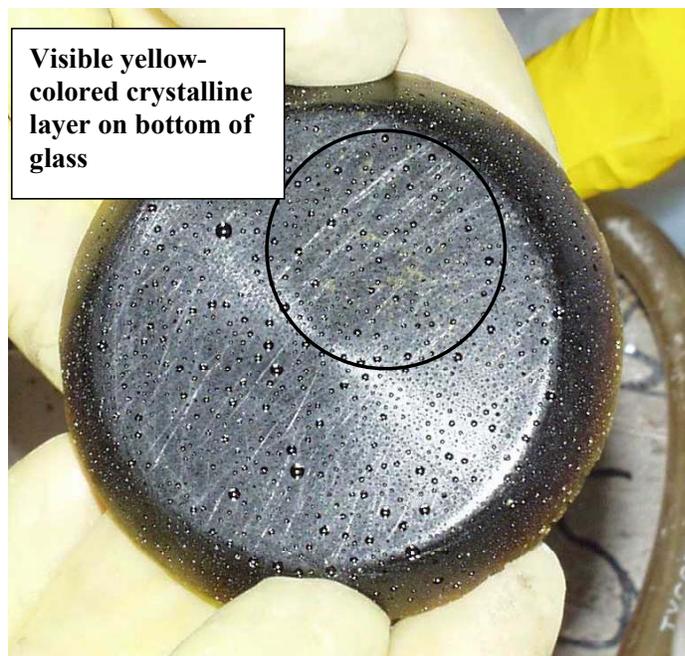
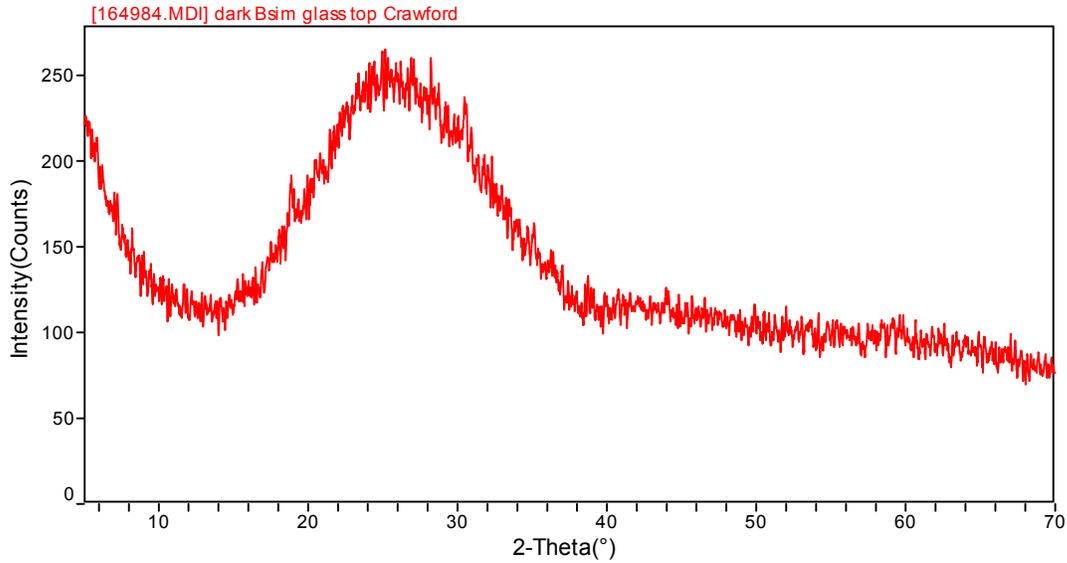
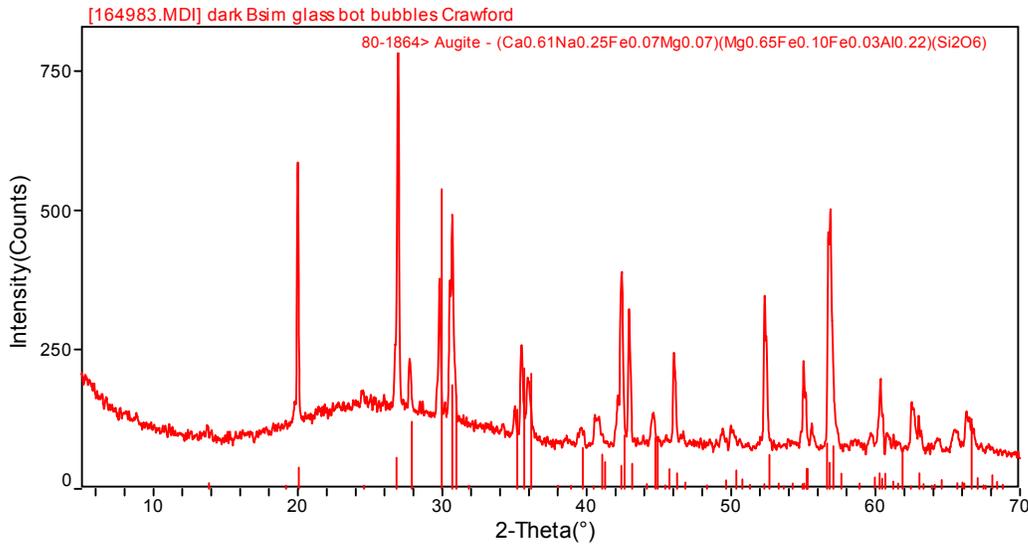


Figure 27. Top Surface XRD Pattern from Test #9 Glass Made in Modified Deltech Furnace and Bottom Surface XRD Pattern from Test #9 Glass Made in Modified Deltech Furnace

(A) No crystalline structure identified in XRD pattern.



(B) Bottom surface XRD pattern indicating presence of augite.



Crystalline Phase Identification

All of the crystalline phases identified in these tests are in the pyroxene family of crystals [17]. Table 12 summarizes the crystal identification of each test sample. The empirical formula for each crystal is also shown in Table 12. Each crystal structure contains 3 different sites (M1, M2 and T). The oxidation state and amount of each element populated in the various crystalline sites were determined by XRD studies of pyroxene minerals by Oberti and Caporuscio [18]. The x-ray diffraction pattern for clinopyroxene (#80-1862) is very similar to the x-ray diffraction pattern for augite (#80-1864). Both crystalline phases are rich in Ca and Mg. Clinopyroxene contains chromium and trace Fe^{3+} with some aluminum substituted for Si in the silicate component ($\text{Si}_{1.97}\text{Al}_{0.03}\text{O}_6$). Augite contains ~ 6X more reduced Fe^{2+} (0.17) than Fe^{3+} (0.03). This abundance of reduced iron in the augite crystals observed in the crucible indicates the possible role of redox in the crystal formation. It should be noted that most of the glass former mineral source of iron added to the glass formulation is in the form of oxidized iron, as Fe_2O_3 (See Table 9). All of the crucible tests were conducted without any added sugar in the glass formulation.

All crucible melts in this study used the 3.5" height 600-mL Pt/Au crucibles shown previously in Figure 2. The final glass pieces produced had nominal thicknesses of ~ 1.2 cm (See previous Equation 2 calculations). It can be shown that redox kinetics in a glass forming melt are ultimately controlled by the diffusion of oxygen into the melt and that the mechanism for oxygen diffusion through a silicate melt involves the retention of oxygen as molecular oxygen that moves through channels or holes within the melt structure [19]. Schreiber et al. report nominal oxygen diffusion times through ~ 7 mm glass melts to be on the order of 4 hours with glasses melted at 1150 °C in ambient air. Thus it could be possible that the bottom-forming crystals observed in this work are related to the lack of complete oxygen diffusion within the glass melt. However, the rapid-cooled radioactive AZ-102 glass that was melted for the same 4-hour time period (See Test #3 results) did not produce the bottom-forming crystals. This single test result suggests that the bottom-forming crystal formation is more likely a result of the slow simulated canister cooling (which did produce the bottom-forming crystals) than a result of incomplete oxygen diffusion into the glass melt.

Two comparisons pertaining to the redox state of the glass can be made from the various crucible melts. In the first comparison, if one compares (Test #1 & Test #2) and #7 glasses with either Tests #5 and/or #6 glasses, the increased amount of observable crystalline phase present in Tests #1 & #2 and #7 vs. either Test #5 or #6 could be explained by the presence of relatively high amounts of total organic carbon in the Tests #1, #2 and #7 feeds. No organic carbon was present in the oxidized VSL simulants tested in Tests #5 and #6.

A second comparison can be made between the Test #7 glass vs. the next two Tests #8 and Test #9 glasses. All of these Tests #7, #8 and #9 used the revised, high organic carbon-containing AZ-102 simulant. Comparing the amount of observable crystals

present on these glasses, it appears that less crystalline phase was produced in this series going from Test #7 (most visible crystalline phase), to Test #8 (lesser amount of visible crystalline phase), to Test #9 (trace amounts of visible crystalline phase) glasses. One possible explanation for this observation is that more ambient oxygen was made available during these crucible vitrification tests in going from Test #7 (sealed quartz crucible) to Test #8 (open crucible in static benchtop furnace) to Test #9 (modified sealed quartz crucible to allow more air flux). Further testing with the SRTC-developed AZ-102 simulant would be required to confirm these preliminary observations.

CONCLUSIONS AND PATH FORWARD

Crucible melts with radioactive AZ-102 material, surrogate material provided by VSL and a revised simulant developed at SRTC have resulted in the presence of a crystalline pyroxene silicate (unnamed clinopyroxene and augite) phase on the bottom surface of the glasses. Quench cooling of one of the radioactive AZ-102 melts produced a glass that did not show presence of the crystalline phase and a glass that had elemental characterization that agreed well with target. It should be noted that no sulfur-containing analytes have been identified with the crystalline phases observed nor have any top-forming sulfate salt layers been observed in this work. Devitrification, i.e., crystallization, is an important consideration in the processing and performance of nuclear waste glasses. While some crystalline formation in glasses has reported little or no effect on leachability, other crystals can increase leachability leading to poorer overall durability [20,21]. Because the glasses produced with the radioactive AZ-102 were not found to be amorphous like previous glass melts produced with Env. A (AN-103) and Env. C (AN-102) studies [11-12], the glasses produced in the current study were not submitted for full characterization and product testing. This study involved mainly investigation of the identification and formation under various conditions of the pyroxene crystals on the glass.

Identification of a reduced state pyroxene crystal in these glasses could indicate a reduced glass product. This could explain why glasses made with radioactive feed and simulant feed containing representative levels of organic carbon, contain up to 5 vol% of crystalline phase. However, similar tests with simulants not containing any organic carbon produced glasses with relatively little observable crystalline phase. A series of tests with AZ-102 simulant that contained representative dissolved organic carbon, in configurations that allow more air contact during melt and cooling also support the apparent relationship between the redox of the glass and the presence of the crystalline secondary phase – as the air flux was increased, lesser amounts of visible crystalline phase was observed on these glasses.

All of our testing has been carried out in 95% Pt/ 5% Au crucibles. Platinum material has been observed in microscopy data from powdered glass samples and glass shards containing the secondary crystalline phase. Further testing involving various crucible materials would need to be performed to explore the role of platinum in relation to the crystal formation.

The general conclusions from these studies are:

- redox appears to affect the amount of augite crystal that forms because tests using increasing amount of air flux show decreasing amounts of crystal formation
- platinum may act as a nucleating agent but does not influence the amount of crystals that form
- because of the redox dependency of the crystallization, simulants containing representative organics must be used to accurately duplicate the crystallization effects shown with the radioactive AZ-102 feed
- simulants do appear to approximate the behavior of the actual waste when the simulants are prepared with representative organics that are analyzed to be in the actual radioactive AZ-102 supernate

PATH FORWARD: Results from these tests have been categorized as ‘new discovery’ findings in the study of the radioactive AZ-102 crucible scale vitrification. Accordingly, the RPP has decided to attempt reformulation of the AZ-102 glass-forming recipe and to pursue further simulant testing with crucible and melter scale studies using an RPP ‘validated’ or ‘approved’ AZ-102 simulant material. These tests would involve the VSL/CUA and GTS-Duratek. The goal of reformulation with the AZ-102 feed would be to produce an amorphous glass upon simulated canister cooling that could be then submitted for full compositional characterization and product testing including PCT and vapor hydration testing. The results presented in this report for the actual radioactive tests and the surrogate or simulant tests give credibility to the importance of a rigorous surrogate development program to support the pretreatment and vitrification Processes in the WTP.

Upon completion of reformulation and simulant testing, the remaining pretreated radioactive AZ-102 concentrated supernate (currently stored at SRTC) will be vitrified in crucible melts at SRTC to produce glass for product testing and analysis. New Task Specifications are currently in development by Bechtel/Washington Group and SRTC personnel to address this proposed future testing with the AZ-102 sample [22,23].

QUALITY CONTROLS AND QUALITY ASSURANCE

QC and QA programs applied to the testing described in this technical report include SRTC procedures for control of measurement and testing equipment (M&TE), tracking of radioactive samples, control of laboratory notebooks, and routine ADS QA and QC [24-26]. The QA program applied by SRTC for preparation and analysis of the AZ-102 glass sample complies with the requirements of NQA-1 [5].

Analytic standards were required for all analyses performed for this study. Use of these standards is part of routine ADS QA and QC, and are part of the procedures in **Manual L16.1** for the operating the analytical instrument.

All M&TE used to perform the evaporation and vitrification experiments was used within the specified calibration period. Calibrations were verified as required for each mass balance instrument. A record of the calibration was routinely maintained in the logbook designated for that piece of equipment.

All personnel who performed steps of the evaporation and vitrification testing were trained on the ITS procedure for operating the evaporation apparatus and furnace. In addition, they were trained on calibrating and operating equipment used in these steps. Training records were maintained for all personnel working on this project.

All laboratory data obtained in the tasks described in this technical report are included as permanent record in Charles L. Crawford's WSRC laboratory notebooks: **WSRC-NB-98-00196** and **WSRC-NB-99-00182**. Associated data to these two WSRC laboratory notebooks are also kept as permanent record in a three-ring binder labeled as: **LAW Envelope B, Sample AZ-102, Vitrification and Product Testing, Charles L. Crawford.**

The contents of the report were verified by document review in accordance with the QA Plan Checklist provided in section IX of the Task Technical and QA Plan [5].

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19. H. D. Schreiber, “Redox Kinetics and Oxygen Diffusion in a Borosilicate Melt”, **Physics and Chemistry of Glasses**, Vol. 27, No. 4, August 1986, pp. 152 – 177.
20. C. M. Jantzen and D. F. Bickford, “Leaching of Devitrified Glass Containing Simulated SRP Nuclear Waste”, **Mat. Res. Soc. Symp. Proc.** Vol. 44, 1985, Materials Research Society, pp. 135 – 146.
21. B. J. Riley, J. A. Rosario and P. Hrma, “Impact of HLW Glass Crystallinity on the PCT Response”, **PNNL-13491**, April 2001.
22. AZ 102 (Envelope B) LAW Vitrification, Product Testing, Contract # DE-AC27-99RL14047, **Document 24590-LAW-TSP-RT-01-024, Rev A**, A. Sidibe and J. Harbour, RPP-WTP Project, Draft in progress, November 2001.
23. AZ 102 (Envelope B) LAW Regulatory Analyses, Contract # DE-AC27-99RL14047, **Document 24590-LAW-TSP-RT-01-025, Rev A**, A. Sidibe and J. Harbour, RPP-WTP Project, Draft in progress, November 2001.
24. Westinghouse Savannah River Company, “**WSRC 1Q Quality Assurance Manual**,” **Manual 1Q**, current revision.
25. Westinghouse Savannah River Company, “**WSRC L1 Savannah River Technology Center Procedures Manual**,” current revision.
26. Westinghouse Savannah River Company, “**WSRC L16.1 Analytical Development Section Analytical Operating Procedures Manual**,” current revision.

Appendix A. Data Sheets for Glass Forming Minerals

[1] Kyanite (Al ₂ SiO ₅) 325 Mesh	Kyanite Mining
[2] H ₃ BO ₃ (Technical Granular)	US Borax
[3] Wollastonite NYAD 325 Mesh, Calcium metaSilicate, CaSiO ₃	NYCO Minerals
[4] Fe ₂ O ₃ (Iron III oxide, -325 Mesh)	Alfa Aesar-Johnson Matthey
[5] Li ₂ CO ₃ (Chemetall Foote Co. Tech.gr.	Cyprus Foote Mineral Co.
[6] Olivine (Mg ₂ SiO ₄) 325 Mesh (#180)	UNIMIN Corp.
[7] SiO ₂ (Sil-co-Sil 75)	US SILICA
[8] TiO ₂ (Rutile Airfloated)	Chemalloy
[9] ZnO (Kadox-920)	Zinc Corp. of America
[10] Zircon ZrSiO ₄ (Flour) Mesh 325	American Mineral

**COMPLETE CHEMICAL ANALYSIS ON KYANITE
CONCENTRATES**

	Kyanite (Raw)	Mullite (Calcined)
Ignition loss	.21	--
Alumina	54.00 - 60.06 <i>57.03</i>	54.17 - 60.06
Silica	43.70 - 37.64 <i>40.67</i>	43.73 - 37.84
Iron Oxide	.40 - 1.16 <i>.78</i>	.36 - .90
Titania	.52 - 1.65 <i>1.03</i>	.52 - .98
Lime	.03	.03
Magnesia	.01	.01
Alkalies	.42	.42
	<u>99.98</u>	<u>99.97</u>

Composition - Raw Kyanite $3\text{Al}_2\text{O}_3\text{-}3\text{SiO}_2$
Calcined Kyanite (Mullite) $3\text{Al}_2\text{O}_3\text{-}2\text{SiO}_2$

- Physical Properties - a. Streak - uncolored
b. Hardness - 4 to 7 Mohr's Scale
c. Specific Gravity Kyanite 3.5 to 3.7
Mullite 2.9 to 3.1
d. Lustre - vitreous to pearly
e. Color - dark grey to sandy
f. Particle shape - bladed (elongated)

Pyrometric Cone Equivalent - Cone 36 to 37
The Calcined Kyanite (Mullite) has been completely converted at a temperature of 3000 deg. F by Kyanite Mining Corp.

Mines - Dillwyn, Virginia (East Ridge Mountain)
Dillwyn, Virginia (Willis Mountain)
Kyanite Grinding Plants - East Ridge
Gieseke
Pamplin
Mullite Production & Grinding Plants - Dillwyn
Cullen
East Ridge

TYPICAL SCREEN ANALYSES ON VIRGINIA KYANITE & MULLITE

KYANITE PRODUCTS

35 Mesh %		48 Mesh %		100 Mesh %		200 Mesh %		325 Mesh %	
On	35 - 9.2	On	48 - 10.0	On	100 - 6.9	On	200 - 9.8	On	325 - 10.1
	48 - 21.2		100 - 29.9		150 - 11.1		325 - 19.6		-325 - 89.9
	100 - 41.4		150 - 13.5		200 - 17.6		-325 - 70.6		
	150 - 16.8		200 - 9.3		325 - 23.9				
	200 - 8.7		325 - 10.7		-325 - 39.5				
	325 - 2.5		-325 - 26.6						
	-325 - .1								
Total	<u>99.9</u>		<u>100.0</u>		<u>99.0</u>		<u>100.0</u>		<u>100.0</u>

MULLITE (Calcined Kyanite) PRODUCTS

35 Mesh %		48 Mesh %		100 Mesh %		200 Mesh %		325 Mesh %	
On	35 - 16.9	On	48 - 4.9	On	100 - 5.8	On	200 - 9.0	On	325 - 9.8
	48 - 21.6		100 - 16.1		150 - 11.6		325 - 18.1		-325 - 90.1
	100 - 38.1		150 - 11.6		200 - 16.7		-325 - 72.0		
	150 - 13.2		200 - 10.0		325 - 27.1				
	200 - 6.9		325 - 18.7		-325 - 38.7				
	325 - 2.6		-325 - 36.7						
	-325 - .6								
Total	<u>99.9</u>		<u>98.0</u>		<u>99.9</u>		<u>99.1</u>		<u>99.9</u>



Boric Acid

Technical Granular

Orthoboric Acid
 H_3BO_3
CAS No. 10043-35-3

Product Specification B-0310-U

May 1, 1998

Boric Acid Technical Granular is a free-flowing, white, crystalline product manufactured in the USA by U.S. Borax Inc.

Chemical specification

	Guarantee	
B_2O_3 %	56.25-56.80	: 56.52
Equivalent H_3BO_3 %	99.9-100.9	
SO_4 ppm	≤ 350	
Cl ppm	≤ 18	
Fe ppm	≤ 6	

Sieve specification

U.S. Standard Sieve No.	% Retained Guarantee
20	≤ 2.0

Note:

All data in the above specifications are determined by U.S. Borax analytical methods.

Packaging

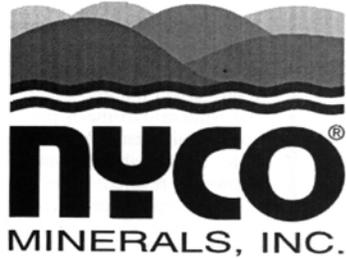
Boric Acid Technical Granular is available in bulk, in 2500 lb. IBCs and in 50 lb. multiwall paper sacks.



Issued by:
U.S. Borax Inc.
26877 Tourney Road
Valencia, CA 91355-1847
USA

Wollastonite = Calcium Metasilicate, CaSiO_3

Wollastonite



**To learn more
 about the
 benefits of
 wollastonite,
 contact:**

NYCO MINERALS INC.
 124 Mountain View Dr.
 P.O. Box 368
 Willsboro, NY 12996-0368
 Phone: 518-963-4262
 Fax: 518-963-1110

Key Benefits and Properties

Production Benefits of Using Wollastonite in Sanitaryware*

- Shrinkage maintained to comply with mold standards
- Reduced vitrification temperature
- Increased impact resistance
- Increased drying temperature
- Reduced drying time
- Reduced HF emissions
- Improved fired strength

*wollastonite with a very high aspect ratio

Typical Physical and Chemical Properties of Wollastonite

Appearance	White
Particle Shape	Acicular
Molecular Weight	116
Specific Gravity	2.9
Refractive Index	1.63
pH (10% slurry)	9.9
Water Solubility (g/100cc)	0.0095
Density (lbs./solid gallons)	24.2
Mohs Hardness	4.5
Coefficient of Expansion (mm/mm/°C)	6.5×10^{-6}
Melting Point (°C)	1540

Chemical Composition

CaO	47.5
SiO ₂	51.0
Fe ₂ O ₃	0.4
Al ₂ O ₃	0.2
MnO	0.1
MgO	0.1
TiO ₂	0.02
L.O.I. (1000°C)	0.68

ALFA Aesar
A Johnson Matthey Company

MATERIAL SAFETY DATA SHEET

Alfa Aesar (A Johnson Matthey Company)
Johnson Matthey Catalog Company, Inc.
30 Bond Street
Ward Hill, MA 01835-0747
Emergency Phone-(978) 521-6300
CHEMTREC-(800) 424-9300
Web Site: www.alfa.com

SECTION 1-IDENTIFICATION

Product Code: 12375 Revision Date: 5/14/96
Product Name: Iron (III) oxide
Synonyms: Ferric oxide
Red iron oxide
Iron red
Iron sesquioxide

Chemical Family: Metal oxide
CAS#: 1309-37-1
Molecular Formula: Fe₂O₃

SECTION 2-INGREDIENTS

Chemical: Iron (III) oxide

CAS#	%	PEL	TLV
1309-37-1	100	10mg Fe/m ³ -fume	5mg Fe/m ³ -dust/fume

SECTION 3- PHYSICAL DATA

Boiling Point: Not applicable
% Volatiles: Not applicable
Solubility in Water : Insoluble
Specific Gravity (H₂O=1): 5.24
Freezing/Melting Point: 1565°C (decomposes)
Evaporation Rate (butyl acetate=1): Not applicable
Vapor Density (air=1): Not applicable
Vapor Pressure : Not applicable
Appearance and Odor: Reddish-brown powder; odorless
Other: No data

SECTION 4-FIRE AND EXPLOSION HAZARD DATA

Flash Point:(°F) Not applicable
Flammable Limits in Air, % by volume: Lower Not applicable
Upper Not applicable
Autoignition Temperature: No data

ALFA Aesar
A Johnson Matthey Company

ALFA Aesar
A Johnson Matthey Company



Cyprus Foote Mineral Company
 Silver Peak Operations, Hwy. 285
 Silver Peak, Nevada 89047
 (702) 937-2222
 FAX (702) 937-2250

CERTIFICATE of ANALYSIS
 LITHIUM CARBONATE
 (CRYSTAL)

ANALYSIS
 DATE: 04/08/95

CHEMICAL ANALYSIS	LOT NO. 503-14 %	REQUIRED SPEC'S %
Li2CO3	99.2	MIN 99.0
Cl	0.017	
SO4	0.12	MAX .40
H2O	0.02	
Ca	0.045	
Mg	0.007	
Na	0.09	
K	0.037	
B	0.03	
Fe2O3	0.001	MAX .004
Insol	0.018	
LOI	0.65	
Reflectance: Blue	93	
Green	93	
SIEVE ANALYSIS		
+10	0.0	MAX 0.0
+20	0.3	MAX 1.5
+30	0.8	
+40	2.8	
+60	19.1	
+100	29.0	
+140	16.0	
+200	10.9	
-200	21.1	MAX 60.0

WE CERTIFY THAT THIS
 LOT MEETS THE MINIMUM
 REQUIREMENTS FOR SP01000. Rev 1.

David H. [Signature]
 QA ANALYST DATE: 4/10/95

FOR SHIPPING USE ONLY
 CUSTOMER RAY SCHUMACHER
 CUSTOMER PO#: VERBAL
 CUSTOMER CODE#: _____
 CFM ORDER #: 1003490



TECHNICAL DATA

**OLIVINE Refractory Grades
HAMILTON, WA**

CHEMICAL ANALYSIS

Mean Values. These Do Not Represent A Specification.

		Mean Percent by Weight
Magnesium Oxide	(MgO)	48.01
Silicon Dioxide	(SiO ₂)	42.52
Iron Oxide	(Fe ₂ O ₃)	7.68
Calcium Oxide	(CaO)	0.02
Chromium Oxide	(Cr ₂ O ₃)	0.13
Aluminum Oxide	(Al ₂ O ₃)	0.19
Potassium Oxide	(K ₂ O)	.01
Sodium Oxide	(Na ₂ O)	.02
Nickel Oxide	(NiO)	.37
Loss on Ignition	(LOI)	1.05

ORDERING INFORMATION

Shipping Point: HAMILTON, WA

Availability: BULK, 50 LB., 100 LB., AND BULK BAGS
TRUCK AND RAIL



FOR PRODUCT INFORMATION AND CUSTOMER SERVICE:
U.S. and CANADA 800-243-9004 • FAX 800-243-9005
WORLDWIDE 203-966-1306 • FAX 203-972-1378

UNIMIN CORPORATION

Silica Sand • Ground Silica • Fofdepar • Naphelino Syonite • High Purity Quartz • Olivine • Microcrystalline Silica • Dentonite Clay • Dolomite

GRADE NUMBERS INDICATE RELATIVE VALUES ON RESULTS. THEY ARE NOT A SPECIFICATION OR WARRANTY OF PERFORMANCE.

HEALTH HAZARD WARNING: Prolonged inhalation of dust associated with the materials described in this data sheet can cause delayed lung injury. This material contains Nickel. IARC has determined Nickel compounds are carcinogenic to humans and the National Toxicological Program (NTP) has determined that Nickel may reasonably be anticipated to be a carcinogen. Avoid creating dust when handling, using or storing. Follow OSHA or other applicable governmental Safety and Health Standards. Current Material Safety Data Sheet containing safety information is available and should be consulted before usage.

Notice: While information contained herein is correct to the best of our knowledge, Unimin Corporation hereby disclaims any warranties as to the accuracy of the same. Recommendations or suggestions are made without guarantee or representation as to result, since conditions of use are beyond our control. All materials are sold to Unimin Corporation standard terms and conditions of sale and on the condition that buyer shall make his own tests to determine the suitability of such product for buyer's purpose. No statement contained herein shall be construed as a recommendation to infringe any patent.

ham-raf (4/97)

Olivina/Olivino Containing

SIL-CO-SIL[®] GROUND SILICA FROM U.S. SILICA COMPANY

New Name	75				63		53	
	200 Mesh	200 Mesh	200 Mesh	200 Mesh			270 Mesh	270 Mesh
Old Name	Pacific, MO	Columbia, SC	Mill Creek, OK	Berkeley Springs, WV			Berkeley Springs, WV	Mill Creek, OK
Mine								
Sieve Analysis								
Cum. % + 100 Mesh	Trace	Trace	Trace	0.1			0.1	Trace
Cum. % + 200 Mesh	1.6	1.5	1.5	0.7			0.3	0.5
Cum. % + 325 Mesh	12.0	13.0	13.0	12.0			6.0	7.0
Particle Size								
Median (Microns)	17.0	16.0	15.0	16.0			15.0	12.0
Average (Microns)	6.0	6.0	6.0	6.0			5.5	5.5
Specific Surface Area, (cm ² /g)	3800	3800	3800	3800			4200	4200
Oil Absorption, (lbs/100 lbs)	20.5	22.0	20.0	24.0			25.0	21.0
Hegman Grind	1	1	1	1			2½	3
Apparent Density, (lbs/cu. ft.)								
Bulk Density, Tapped	93	94	93	94			90	89
Bulk Density, Untapped	55	57	55	58			56	51
Optical Properties								
Reflectance, Green Iris	88.0	86.0	88.0	85.0			85.0	89.0
Reflectance, Blue Iris	85.0	82.0	87.0	82.5			83.0	87.0
Reflectance, Amber Iris	89.0	87.5	90.0	88.0			89.0	90.0
Yellowness	.047	.055	.035	.060			.060	.033
Brightness (457 μm)	86.0	84.0	86.0	84.0			84.0	87.0
pH	7.2	6.0	7.0	6.8			6.8	7.2
Chemical Analysis, %								
SiO ₂	99.7	99.5	99.7	99.6			99.6	99.7
Fe ₂ O ₃	.018	.025	.020	.025			.025	.020
Al ₂ O ₃	.100	.200	.090	.100			.100	.090
TiO ₂	.012	.035	.012	.020			.020	.012
CaO	.01	<.01	.035	.015			.015	.035
MgO	<.01	<.01	<.01	<.01			<.01	<.01
L.O.I	.145	.150	.140	.200			.200	.140

ASTM C-371-56
SEDIGRAPH 50% Point ASTM C-958
Fisher Subsieve ASTM B-330
Derived from Fisher avg. particle size (see above)
ASTM D-1483

ASTM D-1210
U.S. Silica
HunterLab Colorimeter ASTM E-306
Photovolt
5% Slurry
ASTM C-146-72



ZINC CORPORATION OF AMERICA

300 FRANKFORT ROAD
MONACA, PENNSYLVANIA 15061
(412) 774-1020

**KADOX-920
ZINC OXIDE**

DESCRIPTION

KADOX-920 is a high purity French Process zinc oxide, providing a surface area and reactivity between KADOX-911 and KADOX-930. KADOX-920 is also available in coated (KADOX-920C), pelleted (KADOX-920P), and granular (KADOX-920G) forms.

USES

RUBBER — KADOX-920 is used extensively in various rubber products, such as mechanical goods, insulated wire, footwear, and tires, where uniform activation, moderate reinforcement, and good dispersion are desired.

OTHER USES — KADOX-920 is used in the production of ceramics, rayon, resinates, textiles, zinc chromates, and phosphate solutions where a zinc oxide of high purity is required.

TECHNICAL DATA

Representative Physical Properties

Mean Particle Size (microns)	0.21
Surface Area (sq. meters/gram)	5.0
Specific Gravity	5.6
Apparent Density (lb./ft. ³)	35.
Through 325 Mesh	99.99%
Specifications	ASTM D-79 ASTM D-4295

**Representative
Chemical Properties**

ZnO	99.8%
PbO001%
CdO005%
CuO	<.0005%
MnO	<.0005%
Fe ₂ O ₃001%
H ₂ O Soluble Salts ..	.02%

The information hereon has been compiled from sources which we believe to be reliable, but we assume no responsibility or liability for its accuracy or for the result of any application made of any information contained herein, nor do we assume any liability for infringement of any patent which may result from the application of such information.



American Minerals, Inc.

901 E. Eighth Avenue, Suite #200
 King of Prussia, Pennsylvania 19406

ZIRCON
Zirconium Silicate

ZIRCON

Zircon Sand and Flour for all applications. Large stockpiles maintained assuring consistent high quality and reliable supply of different grades and grinds for various industries.

APPLICATIONS

- Steel
- Refractory
- Foundries
- Ceramics
- Glass
- Chemical
- Abrasive

SPECIFICATION

Chemical:		
<u>Constituent -</u>	<u>Typical Range - Wt. %</u>	
ZrO ₂ (+HfO ₂)	66.0% Typ.	65.0 Min.
Fe ₂ O ₃	0.06 - 0.09	
TiO ₂	0.07 - 0.14	
Al ₂ O ₃	0.1 - 0.4	
SiO ₂	32.0 - 32.5	
Free Silica	0.01 - 0.2	
U + Th	400 ppm.	500 ppm. Max.

Physical:			
Sand:	<u>Typical</u>	<u>'B' Grade</u>	<u>'C' Grade</u>

Data:

		40	0	0
		50	0	1.0
		70	0.20	28.0
		100	8.00	55.0
Specific Gravity	4.6-4.8	140	50.00	14.0
Bulk Density	170-180 lbs./ft. ³	200	41.00	1.5
L.O.I.	0.15-0.25	270	1.30	0.5
Melting Point	2200°C	PAN	0	0
Hardness (MOHS)	7.5			
Angle of repose	30°		AFS 108-115	AFS 68-74

Flour:		<u>Typical</u>	
	<u>Grind</u>	<u>Wt. %</u>	<u>Mesh</u>
	200	95	-200
	325	95	-325

Special Grinds Available

'B' Grade and Flour available as Calcined Zircon.

Appendix B. Comparison of LAW Glass Compositions

**Table B-1. Target Compositions for LAW Glasses for Env. A, AN-103;
Env. C, AN-102 and Env. B, AZ-102.**

Major Element	Env. A* (AN-103) (wt% oxide)	Env. C** (AN-102) (wt% oxide)	Env. B*** (AZ-102) (wt% oxide)
Al ₂ O ₃	6.2	6.1	6.1
B ₂ O ₃	8.9	10.1	10.0
CaO	2.0	6.4	6.7
Cr ₂ O ₃	<0.1	<0.1	0.1
Fe ₂ O ₃	7.0	6.5	5.3
K ₂ O	0.6	0.1	0.2
Li ₂ O	<0.1	2.7	5.9
MgO	2.0	1.5	3.0
Na ₂ O	20.0	11.8	5.0
SiO ₂	44.7	46.7	48.9
TiO ₂	2.0	1.1	1.4
ZnO	3.0	3.0	3.2
ZrO ₂	3.0	3.0	3.2
Cl	0.3	0.1	<0.1
F	<0.1	0.1	<0.1
P ₂ O ₅	<0.1	0.1	<0.1
SO ₃	0.1	0.4	0.8
SUM:	100	100	100

*Env. A, AN-103 data from “Crucible-Scale Active Vitrification Testing
Env. A, Tank 241-AN-103”, WSRC-TR-2000-00322, SRT-RPP-2000-
00021, Rev. 1, June 15, 2001.

**Env. C, AN-102 data from “Crucible-Scale Active Vitrification Testing
Env. C, Tank 241-AN-102”, WSRC-TR-2000-00371, SRT-RPP-2000-
00022, Rev. 0, June 15, 2001.

*** Env. B, AZ-102 data from this report, See Table 9 in text.

Appendix C. Temperature Profiles for Crucible Melts

Figure C-1. Tests #1 and #2 Temperature and Condensate vs. Time Graph
Note that the actual data shown is from Test #1 conducted week of 7/5/00.
Test #2 data was replicated later during week of 7/10/00.

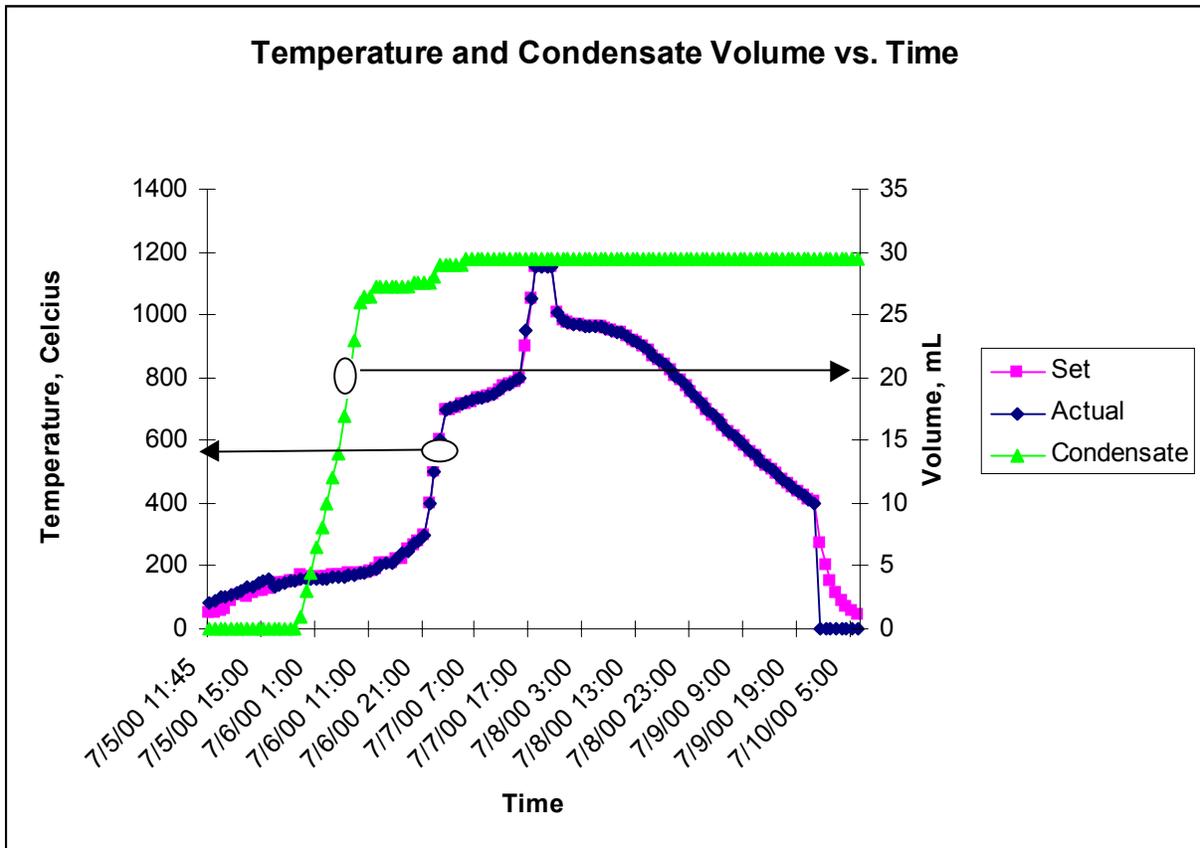


Figure C-2. Test #3 Temperature vs. Time Graph
Note that the heat-up and melt time profile is similar to Tests #1 and #2, followed by a relatively rapid cooling of the glass.

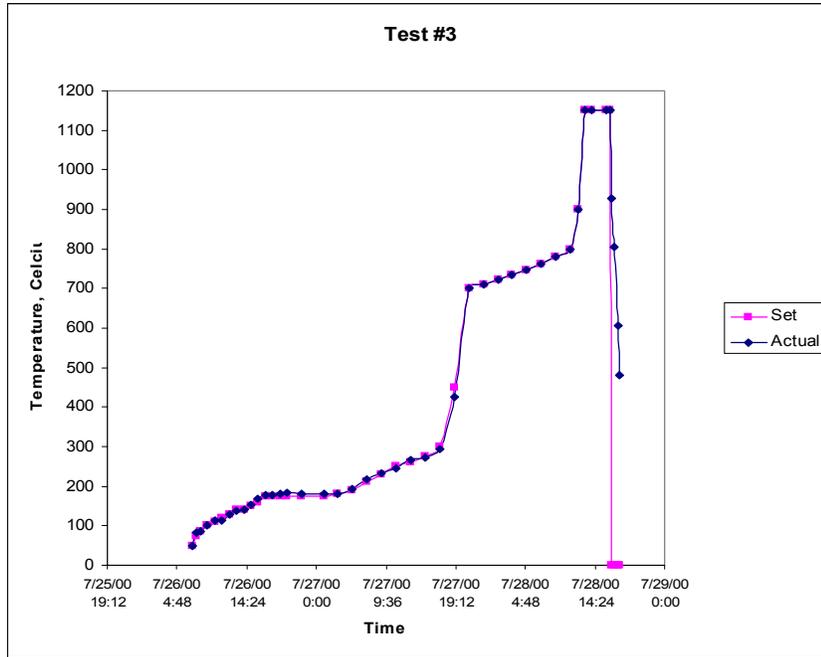


Figure C-3. Test #4 Temperature vs. Time Graph
Note that the product radioactive glass from Test #3 was rapidly heated up to melt temperature of 1150 °C, followed by a canister cooling similar to the canister cooling applied in Tests #1 and #2.

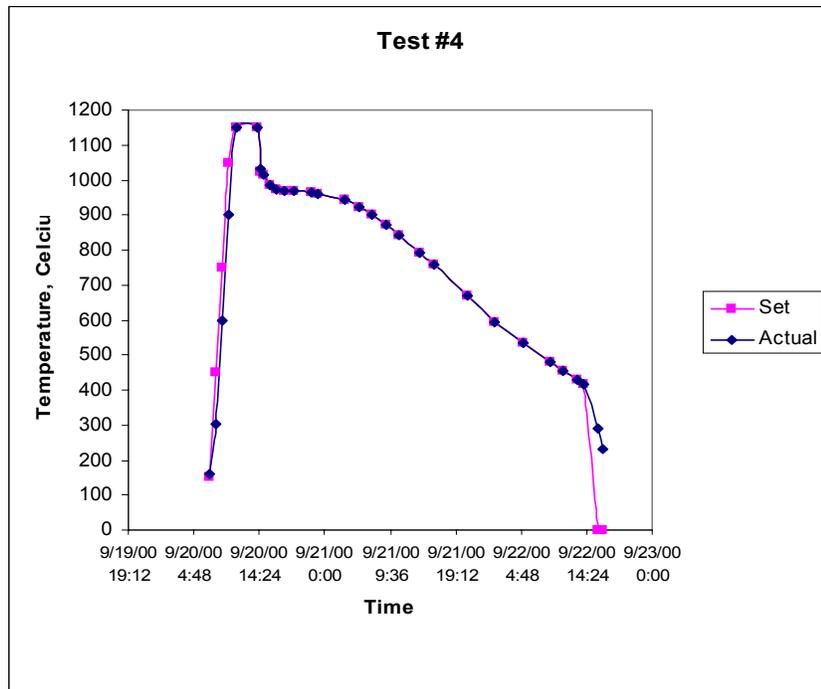


Figure C-4. Test #5 Temperature vs. Time Graph for VSL Powder Simulant LAWB53SUR2. This Test #5 was performed with the same temperature vs. time profile that was used in replicate Tests #1 and #2.

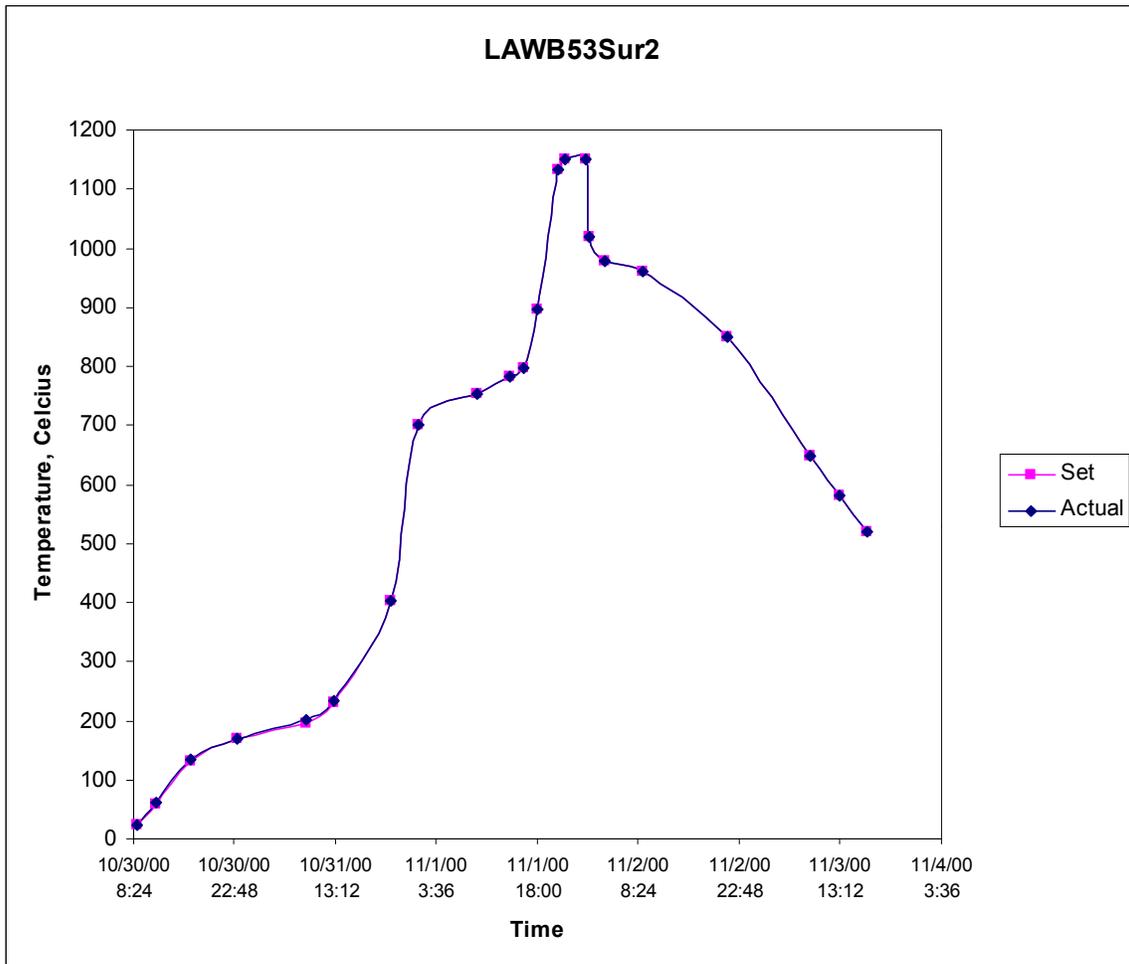


Figure C-5. Test #6 Temperature vs. Time Graph for VSL Glass Simulant LAWB53SUR1. Glass sample was remelted in sealed quartz Deltech furnace and allowed to cool via simulated canister cool curve. The yellow data trace indicates the percentage full power of the Deltech furnace during the heat, melt and cool regions.

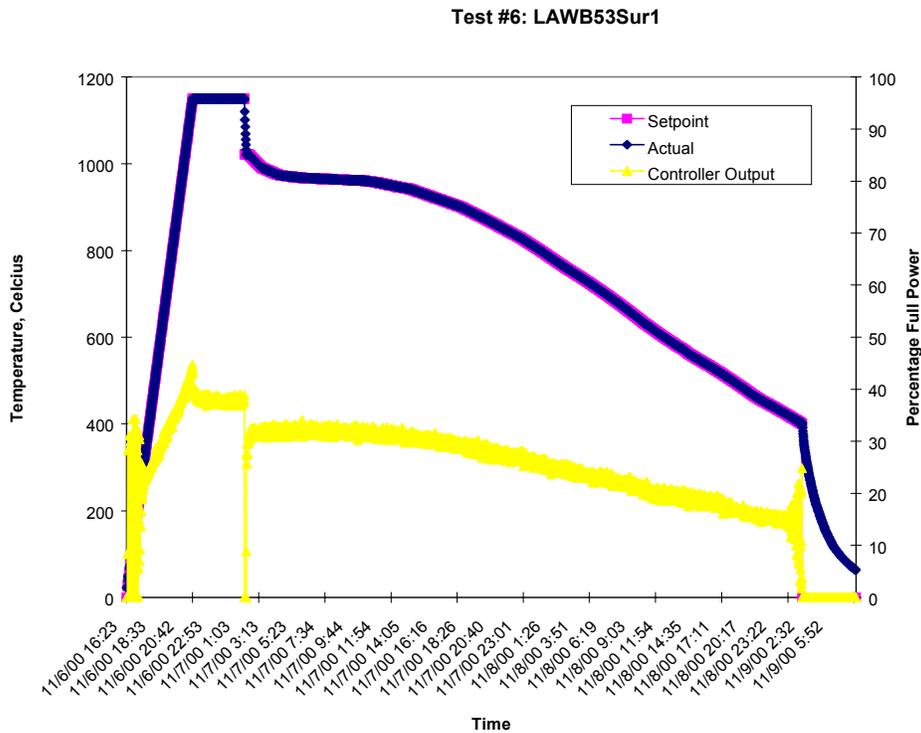


Figure C-6. Test #7 Temperature and Condensate vs. Time Graph for Crucible Melt of AZ-102 Simulant.

- Note:** (1) the temperature vs. time profile for this Test #7 was similar to the initial tests with radioactive AZ-102 feed in Tests #1 and #2.
(2) the amount of condensate collected (>50 mL) is greater than the amount of liquid feed used, indicating condensation of moist ambient air flowing through offgas system.
(3) this Test #7 used the same identical temperature vs. time profile as was used in Test #8.

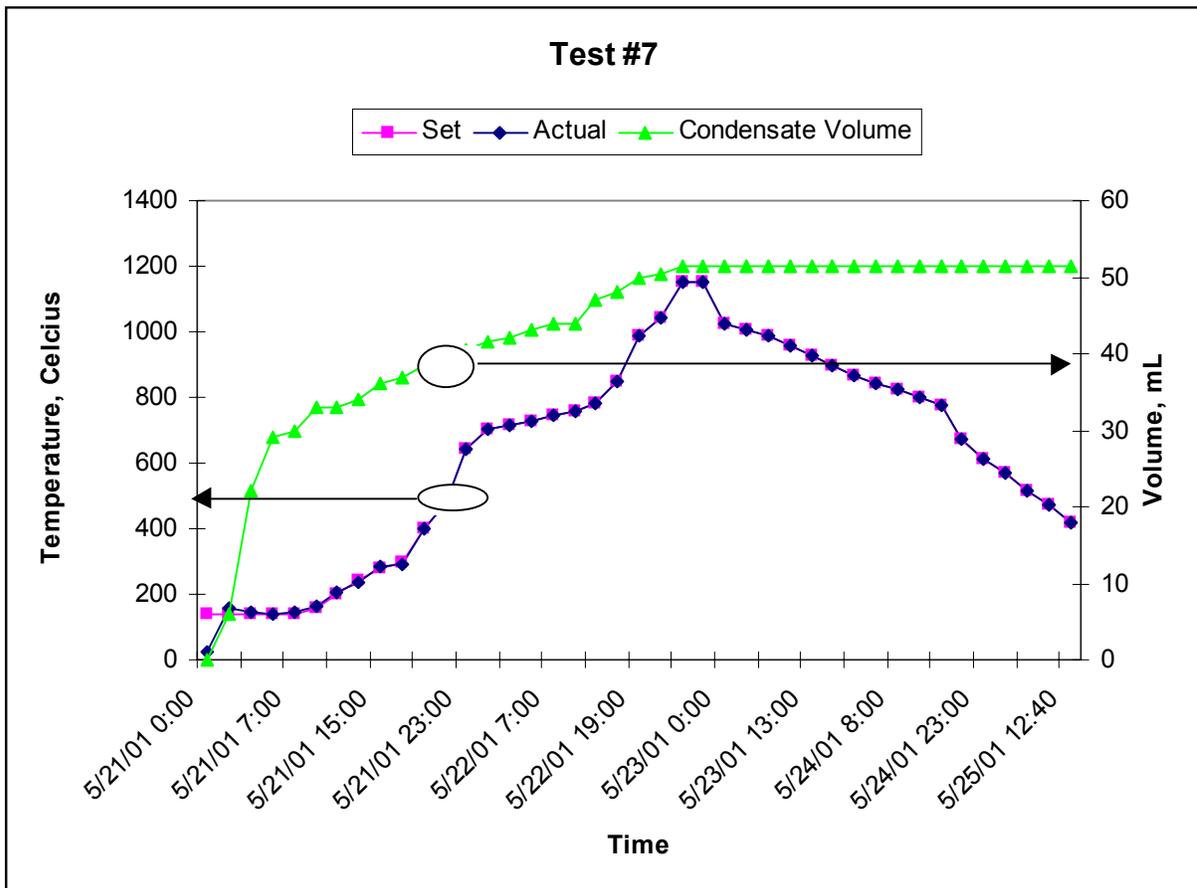
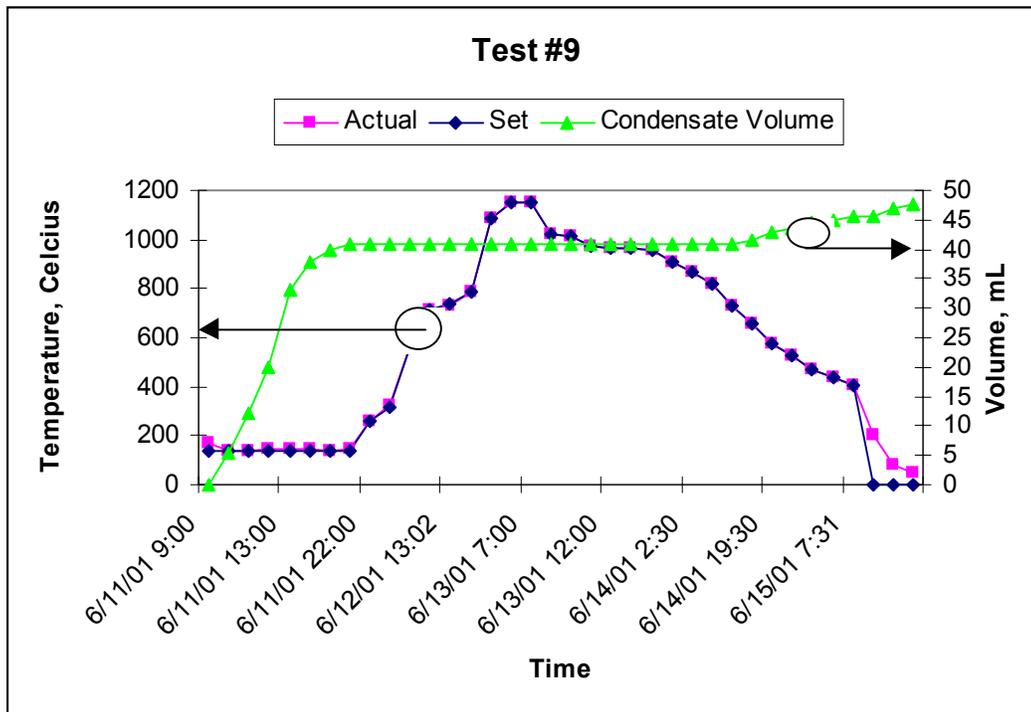


Figure C-7. Test #9 Temperature and Condensate vs. Time Graph for Crucible Test Performed in Modified Deltech Sealed Quartz Furnace with AZ-102 Simulant.

Note the amount of condensate collected was near target (~ 40 ml) until latter part of testing (6/14/01) when the indicating color of the modified silica gel moisture trap had changed from deep blue to pink (See Text).



Appendix D. AZ-102 Simulant Information

Table D-1. Sources of Analyte Analyses for AZ-102 Simulant Development

Information Received from Charles Crawford.							
Envelope B, AZ 102 Radioactive Evaporator Feed				Env. B, AZ 102 EVAPORATOR Concentrate		CONCENTRATION FACTOR	
Cs-137 (uCi/mL)		0.6474		1.2307		1.9	
Co-60 (uCi/mL)	<	0.0016		0.0023			
Ba-154 (uCi/mL)	<	0.0068		0.0011			
Ba-155 (uCi/mL)	<	0.0090		0.0017			
Tc-99 (uCi/mL)				0.0094			
Tc-99 (ug/L)		0.0076		5.9500			
Pu-238 (dpm/mL)		485.9912		(not measure)			
Pu-239/240 (dpm/mL)		1049.0600		(not measure)			
St-90 (uCi/mL)				1.2365			
ICP-MS (ug/L)							
mass 230	<	0.0951		0.6500			
231	<	0.0951		0.6500			
232		37.2929		0.6500			
233	<	0.0951		0.6500			
234	<	0.0951		0.6500			
235		27.5807		34.9000			
236	<	0.0951		0.6500			
237		136.2019		199.9500		1.5	
238		2338.5361		3625.0000		1.6	
239	<	0.0502		75.1600			
240	<	0.0502		4.9250			
241	<	0.0502		0.6500			
242	<	0.0502		0.6500			
243	<	0.0502		0.6500			
244	<	0.0502		0.6500			
245	<	0.0502		0.6500			
246	<	0.0502		0.6500			
U - (mg/L) by Chemcheck		1.6097		(not measure)			
AA (mg/L)				CONCENTRATE			
K		2683.927		4564.8475		1.8	
Na		50887.827	2.213495847	100816.743		4.39	2.0
As		0.329		(not measure)			
Se		0.407		(not measure)			
Hg	<	0.111		(not measure)			
Carbon (mg/L)							
inorganic (TIC)		8616.108		9396		+/-407	1.1
Organic (TOC)		8344.317		19914		+/-7500	2.4
density (g/mL)		1.097		1.16			
wt% Total solids		12.515		24.945			2.0
wt.% suspended solids	<	0.004		(not measure)			
AL-O2(M)	<	0.020		0.2			10.0
Free OH (M)		0.223		1.052			4.7
CO3		(not measure)		0.533			
IC (mg/L)							
NO3-		13387.996		27305			2.0
NO2-		26147.197		49440			1.9
PO4		245.072		1000			4.1
SO4		13287.200		26300			2.0
Oxalate		2400.535		4625			1.9
Formate	<	111.396		1000			9.0
Cl- (by IC)		66.838		200			3.0
F- (by IC)		868.891		1370			1.6
Cl- (by ISE)		182.024		(not measure)			
F- (by ISE)		863.267		(not measure)			
ICP-ES (mg/L)							
Al		401.846		797.59			2.0
B		1.136		4.55			4.0
Ba		1.342		1.82			1.4
Ca		0.567		66.965			118.1
Cd	<	0.067		2.73			40.8
		(not measure)	(Ce)	286.65			
Co		0.139		4.55			32.6
Cr		532.681		1018.085			1.9
Cu	<	0.067		2.73			40.8
Fe	<	0.067		5.46			81.7
La	<	0.245		10.01			40.8
Li	<	0.045		1.82			40.8
Mg	<	0.022		3.08			138.2
Mn	<	0.022		0.91			40.8
Mo		43.168		79.12			1.8
Na		(not measure)	(Na)	89259.89		3.88	
Ni	<	0.156		6.37			40.8
P		139.412		229.63			1.6
Pb		1.171		25.605			21.9
Si		99.675		56.075			0.6
Sn		7.491		17			2.3
Sr	<	0.022		0.91			40.8
Ti	<	0.045		1.82			40.8
V		0.362		2.73			7.5
Zn		0.434		2.73			6.3
		(not measure)	(Zr)	3.64			
				Average Na Concentration (mg/L) =			
				95036 average		4.13	
				8172 st deviation		0.36	
				9 % RSD			
New Data as of 4/27/01							
TIC				10,588		mg/L	
TOC				5,004		mg/L	
Citrate				6,803		mg/L	
Glycolate				13,550		mg/L	
Formate				7,270		mg/L	
Acetate				1,050		mg/L	
Carbonate				34,030		mg/L	
EDTA				350		mg/L	
HEDTA				<250		mg/L	

Table D-2. Analytical Terms for AZ-102 Simulant Development

Tank AZ102 Supernate Composition							
Component	Molecular weight	Concentration	Units	Concentration	Units	Moles of plus	Moles of Minus
Aluminum	26.98154	798.0	mg/Liter	2.96E-02	Molar		2.96E-02
Ammonia	17.03052	0.0	mg/Liter	0.00E+00	Molar	0.00E+00	
Arsenic	74.9216	0.0	mg/Liter	0.00E+00	Molar		0.00E+00
Barium	137.33	0.0	mg/Liter	0.00E+00	Molar	0.00E+00	
Boron	10.81	0.0	mg/Liter	0.00E+00	Molar	0.00E+00	
Cadmium	112.41	0.0	mg/Liter	0.00E+00	Molar	0.00E+00	
Calcium	40.08	67.0	mg/Liter	1.67E-03	Molar	3.34E-03	
Carbonate	60.0092	46944.2	mg/Liter	7.82E-01	Molar		1.56E+00
Oxalate	88.0196	4625.0	mg/Liter	5.25E-02	Molar		1.05E-01
Cesium	132.9054	0.1	mg/Liter	4.26E-07	Molar	4.26E-07	
Chloride	35.453		mg/Liter	0.00E+00	Molar		0.00E+00
Chromium	51.996	1018.0	mg/Liter	1.96E-02	Molar		3.92E-02
Copper	63.546		mg/Liter	0.00E+00	Molar	0.00E+00	
Fluoride	18.998	1370.0	mg/Liter	7.21E-02	Molar		7.21E-02
Hydroxide	17.00734	0.0	mg/Liter	0.00E+00	Molar		0.00E+00
Iron	55.847		mg/Liter	0.00E+00	Molar	0.00E+00	
Lanthanum	138.9055		mg/Liter	0.00E+00	Molar	0.00E+00	
Lead	207.2		mg/Liter	0.00E+00	Molar	0.00E+00	
Lithium	6.941	0.0	mg/Liter	0.00E+00	Molar	0.00E+00	
Magnesium	24.305	3.1	mg/Liter	1.28E-04	Molar	2.55E-04	
Manganese	54.938	0.0	mg/Liter	0.00E+00	Molar	0.00E+00	
Molybdenum	95.94	79.0	mg/Liter	8.23E-04	Molar		1.65E-03
Neodymium	144.24	0.0	mg/Liter	0.00E+00	Molar	0.00E+00	
Nickel	58.69	0.0	mg/Liter	0.00E+00	Molar	0.00E+00	
Nitrate	62.0049	27305.0	mg/Liter	4.40E-01	Molar		4.40E-01
Nitrite	46.0055	49440.0	mg/Liter	1.07E+00	Molar		1.07E+00
Phosphate	94.97136		mg/Liter	0.00E+00	Molar		0.00E+00
Potassium	39.0983	4555.0	mg/Liter	1.17E-01	Molar	1.17E-01	
Rhenium	186.207		mg/Liter	0.00E+00	Molar		0.00E+00
Rhodium	102.9055	0.0	mg/Liter	0.00E+00	Molar	0.00E+00	
Ruthenium	101.07	0.0	mg/Liter	0.00E+00	Molar	0.00E+00	
Selenium	78.96	0.0	mg/Liter	0.00E+00	Molar		0.00E+00
Silicon	28.0855	56.0	mg/Liter	1.99E-03	Molar		3.99E-03
Silver	107.8682		mg/Liter	0.00E+00	Molar	0.00E+00	
Sodium	22.9898	100820.0	mg/Liter	4.39E+00	Molar	4.39E+00	
Strontium	87.62	0.1	mg/Liter	9.99E-07	Molar	2.00E-06	
Sulfate	96.0576	26300.0	mg/Liter	2.74E-01	Molar		5.48E-01
Tellurium	127.6	0.0	mg/Liter	0.00E+00	Molar		0.00E+00
Titanium	47.88	0.0	mg/Liter	0.00E+00	Molar		0.00E+00
TIC	12.011	9396.0	mg/Liter	7.82E-01	Molar		
Vanadium	50.9415	0.0	mg/Liter	0.00E+00	Molar		0.00E+00
Zinc	65.38	0.0	mg/Liter	0.00E+00	Molar	0.00E+00	
Zirconium	91.22	0.0	mg/Liter	0.00E+00	Molar		0.00E+00
Acetate	59.04462	1050.0	mg/Liter	1.78E-02			1.78E-02
Citrate	189.09618	6803.0	mg/Liter	3.60E-02			1.08E-01
Formate	45.01774	7270.0	mg/Liter	1.61E-01			1.61E-01
Glycolate	75.04206	13560.0	mg/Liter	1.81E-01			1.81E-01
EDTA	288.20824	350.0	mg/Liter	1.21E-03			4.86E-03
Totals						4.51	4.35

Table D-3. Recipe for AZ-102 Simulant Development

AZ-102 Evaporator Concentrate Recipe

Volume of Feed	1000 mL
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In a Hood, in a tared 1 Liter Volumetric Flask

Add the following

	grams	Actual Wt. grams
Water	200	

Transition Metals and Complexing agents

Compounds	Formula	Mass Needed	Actual Wt. grams
Ammonium Nitrate	NH4NO3	0.000	
Barium Nitrate	Ba(NO3)2	0.000	
Boric Acid	H3BO3	0.000	
Cadmium Nitrate	Cd(NO3)2.4H2O	0.000	
Calcium Nitrate	Ca(NO3)2.4H2O	0.395	
Cesium Nitrate	CsNO3	0.000	
Copper Nitrate	Cu(NO3)2.2.5H2O	0.000	
Ferric Nitrate	Fe(NO3)3.9H2O	0.000	
Lanthanum Nitrate	La(NO3)3.6H2O	0.000	
Lead Nitrate	Pb(NO3)2	0.000	
Magnesium Nitrate	Mg(NO3)2.6H2O	0.033	
Manganous Chloride	MnCl2.4H2O	0.000	
Potassium Molybdate	K2MoO4	0.196	
Neodymium Nitrate	Nd(NO3)3.6H2O	0.000	
Nickel Nitrate	Ni(NO3)2.6H2O	0.000	
Potassium Nitrate	KNO3	11.612	
Strontium Nitrate	Sr(NO3)2	0.0002	
Zinc Nitrate	Zn(NO3)2.6H2O	0.000	
Zirconyl Nitrate	ZrO(NO3)2.xH2O	0.000	
Sodium Acetate	NaCH3COO.3H2O	2.420	
EDTA	Na2EDTA	0.452	
Glycolic Acid	HOCH2COOH, 70 wt%	19.632	
Citric Acid	C6H8O7.H2O	7.560	
Sodium Oxalate	Na2C2O4	7.041	
Sodium Chloride	NaCl	0.000	
Sodium Fluoride	NaF	3.028	
Sodium Sulfate	Na2SO4	38.890	

In a separate container mix the following

Compounds	Formula	Mass Needed	Actual Wt. grams
Aluminum trihydroxide	Al(OH)3	2.307	
Sodium Hydroxide	NaOH	19.083	
Sodium meta-silicate	Na2SiO3.9H2O	0.567	
Sodium Formate	HCOONa	10.983	
Sodium Phosphate	Na3PO4.12H2O	0.000	

Add	grams	Actual Wt. grams
Water	300	

Mix thoroughly. Then add this solution to the volumetric flask.

Add	Formula	Mass Needed	Actual Wt. grams
Sodium Carbonate	Na2CO3	82.914	

Mix thoroughly.

Add	Formula	Mass Needed	Actual Wt. grams
Sodium Chromate	Na2CrO4	3.171	
Sodium Nitrate	NaNO3	27.359	
Sodium Nitrite	NaNO2	74.151	
Water		Dilute to Mark	

Add and Mix thoroughly.

Agitate for 24 hours to completely dissolve as much as possible.

Interim Report for Crucible-Scale
Active Vitrification of Waste
Envelope B (AZ-102)

WSRC-TR-2001-00395
SRT-RPP-2001-00153
Rev. DRAFT

Table D-4. Calculations for AZ-102 Simulant Development

Compounds	Moles	Nitrate Moles	Contains [Na]	Moles of Na Needed	Wt % Carbon	Formula	Formula Weight grams	Mass Needed grams	Mass of H2O grams	TOC grams	TIC grams
Aluminum trihydroxide	2.96E-02			2.96E-02		Al(OH) ₃	78	2.307	0.00E+00		
Ammonium Nitrate	0.00E+00	0.00E+00				NH ₄ NO ₃	80.04	0.000			
Barium Nitrate	0.00E+00	0.00E+00				Ba(NO ₃) ₂	261.35	0.000			
Boric Acid	0.00E+00			0.00E+00		H ₃ BO ₃	61.83	0.000			
Cadmium Nitrate	0.00E+00	0.00E+00				Cd(NO ₃) ₂ ·4H ₂ O	308.47	0.000	0.00E+00		
Calcium Nitrate	1.67E-03	3.34E-03				Ca(NO ₃) ₂ ·4H ₂ O	236.15	0.395	1.20E-01		
Cesium Nitrate	4.26E-07	4.26E-07				CsNO ₃	194.91	0.000			
Sodium Chromate	1.96E-02		3.92E-02			Na ₂ CrO ₄	161.97	3.171			
Copper Nitrate	0.00E+00	0.00E+00				Cu(NO ₃) ₂ ·2.5H ₂ O	232.59	0.000	0.00E+00		
Ethylenediaminetetraacetic acid	0.00E+00		0.00E+00	0.00E+00	32.3	Na ₂ EDTA·2H ₂ O	372.24	0.000		0.00	
Ferric Nitrate	0.00E+00	0.00E+00				Fe(NO ₃) ₃ ·9H ₂ O	404	0.000	0.00E+00		
Lanthanum Nitrate	0.00E+00	0.00E+00				La(NO ₃) ₃ ·6H ₂ O	433.03	0.000	0.00E+00		
Lead nitrate	0.00E+00	0.00E+00				Pb(NO ₃) ₂	331.2	0.000			
Lithium Chloride	0.00E+00					LiCl	42.39	0.000			
Magnesium Nitrate	1.26E-04	2.55E-04				Mg(NO ₃) ₂ ·6H ₂ O	256.41	0.033	1.38E-02		
Manganese Chloride	0.00E+00					MnCl ₂ ·4H ₂ O	197.9	0.000	0.00E+00		
Potassium Molybdate	8.23E-04					K ₂ MoO ₄	238.14	0.196			
Neodymium Nitrate	0.00E+00	0.00E+00				Nd(NO ₃) ₃ ·6H ₂ O	438.35	0.000	0.00E+00		
n-Hydroxyethylenediaminetriacetic acid	0.00E+00			0	43.2	HEDTA	278.26	0.000		0.00	
Nickel Nitrate	0.00E+00	0.00E+00				Ni(NO ₃) ₂ ·6H ₂ O	290.81	0.000	0.00E+00		
Potassium Nitrate	1.15E-01	1.15E-01				KNO ₃	101.1	11.612			
Sodium Perhenate	0.00E+00		0.00E+00			NaReO ₄	273.19	0.000			
Rhodium Nitrate	0.00E+00	0.00E+00				Rh(NO ₃) ₃ ·2H ₂ O	324.95	0.000	0.00E+00		
Ruthenium Chloride	0.00E+00					RuCl ₃	207.43	0.000			
Selenium dioxide	0.00E+00			0.00E+00		SeO ₂	110.96	0.000			
Sodium meta-silicate	1.99E-03		3.99E-03			Na ₂ SiO ₃ ·9H ₂ O	284.2	0.567	3.23E-01		
Silver Nitrate	0.00E+00	0.00E+00				AgNO ₃	169.87	0.000			
Strontium Nitrate	9.99E-07	2.00E-06				Sr(NO ₃) ₂	211.63	0.000			
Tellurium Dioxide	0.00E+00			0.00E+00		TeO ₂	159.6	0.000			
Titanium Tetrachloride	0.00E+00					TiCl ₄	189.71	0.000			
Vanadium Pentoxide	0.00E+00			0.00E+00		V ₂ O ₅	181.88	0.000			
Zinc Nitrate	0.00E+00	0.00E+00				Zn(NO ₃) ₂ ·6H ₂ O	297.47	0.000	0		
Zirconyl Nitrate	0.00E+00	0				ZrO(NO ₃) ₂ ·xH ₂ O	249.23	0.000	0		
EDTA	1.21E-03			2.43E-03	32.3	Na ₂ EDTA	372.24	0.45		0.15	
Citric Acid	3.60E-02			1.08E-01	34.3	C ₆ H ₈ O ₇ ·H ₂ O	210.14	7.56	0.65	2.59	
Sodium Acetate	1.78E-02		1.78E-02		17.7	NaCH ₃ COO·3H ₂ O	136.08	2.420	9.60E-01	0.43	
Sodium Bromide	0.00E+00		0.00E+00			NaBr	102.89	0.000			
Sodium Carbonate	7.82E-01	1.56E+00			11.3	Na ₂ CO ₃	105.99	82.914			9.40
Sodium Chloride	0.00E+00	0.00E+00				NaCl	58.44	0.000			
Sodium Fluoride	7.21E-02	7.21E-02				NaF	41.99	3.028			
Sodium Formate	1.61E-01	1.61E-01			17.7	HCOONa	68.01	10.983		1.94	
Glycolic Acid	1.81E-01			1.81E-01	31.6	HOCH ₂ COOH, 70 wt%	76.05	19.632	5.889485748	6.20	
Sodium Nitrite	1.07E+00	1.07E+00				NaNO ₂	69	74.151			
Sodium Oxalate	5.25E-02	1.05E-01			17.9	Na ₂ C ₂ O ₄	134	7.041		1.26	
Sodium Phosphate	0.00E+00	0.00E+00				Na ₃ PO ₄ ·12H ₂ O	380.12	0.000	0.00E+00		
Sodium Sulfate	2.74E-01	5.48E-01				Na ₂ SO ₄	142.04	38.890			
Sodium Nitrate	3.22E-01	3.22E-01				NaNO ₃	84.99	27.359			
Sodium Hydroxide	3.21E-01		3.21E-01			NaOH	40	12.825			
Totals		4.40E-01	4.23E+00			Total		305.536	7.95E+00	1.26E+01	9.40E+00
Target		4.40E-01	4.39E+00								
		0.00E+00	1.56E-01								
Additional NaOH to match Na+	1.56E-01					NaOH	40	6.258			
Total NaOH	4.77E-01					NaOH	40	19.083			
						Total		311.794	7.95E+00		
Measured Density	1.16	grams/mL									
Total Solution Mass	1160	grams/mL									
% Solids	26.19										
Mass of Water	848.21	grams									

Table D-5. Experimental Terms in AZ-102 Simulant Development

Volume	1000 mL						
Compounds	Formula	Formula Wt	Actual Mass. grams	Moles	Water. grams	Solids Mass	Na. Moles
Aluminum Trihydroxide	Al(OH) ₃	78	2.307	0.029576923		2.307	
Ammonium Nitrate	NH ₄ NO ₃	80.04	0	0		0.000	
Barium Nitrate	Ba(NO ₃) ₂	261.35	0	0		0.000	
Boric Acid	H ₃ BO ₃	61.83	0	0		0.000	
Cadmium Nitrate	Cd(NO ₃) ₂ ·4H ₂ O	308.47	0	0	0.000	0.000	
Calcium Nitrate	Ca(NO ₃) ₂ ·4H ₂ O	236.15	0.395	0.001672666	0.121	0.274	
Cerium Nitrate	Ce(NO ₃) ₃ ·6H ₂ O	434.23	0	0	0.000	0.000	
Cesium Nitrate	CsNO ₃	194.91	0	0		0.000	
Sodium Chromate	Na ₂ CrO ₄	161.97	3.171	0.0195777		3.171	0.039
Copper Nitrate	Cu(NO ₃) ₂ ·2.5H ₂ O	232.59	0	0	0.000	0.000	
Ethylenediaminetetraacetic acid	Na ₂ EDTA·2H ₂ O	372.24	0.452	0.00121427	0.044	0.408	0.002
Ferric Nitrate	Fe(NO ₃) ₃ ·9H ₂ O	404	0	0	0.000	0.000	
Lanthanum Nitrate	La(NO ₃) ₃ ·6H ₂ O	433.03	0	0	0.000	0.000	
Lead nitrate	Pb(NO ₃) ₂	331.2	0	0		0.000	
Magnesium Nitrate	Mg(NO ₃) ₂ ·6H ₂ O	256.41	0.033	0.0001287	0.014	0.019	
Manganous Chloride	MnCl ₂ ·4H ₂ O	197.9	0	0	0.000	0.000	
Potassium Molybdate	K ₂ MoO ₄	238.14	0.196	0.000823045		0.196	
Neodymium Nitrate	Nd(NO ₃) ₃ ·6H ₂ O	438.35	0	0	0.000	0.000	
n-Hydroxyethylenediaminetriacetic acid	HEDTA	278.26	0	0		0.000	
Nickel Nitrate	Ni(NO ₃) ₂ ·6H ₂ O	290.81	0	0	0.000	0.000	
Potassium Nitrate	KNO ₃	101.1	11.612	0.114856578		11.612	
Selenium dioxide	SeO ₂	110.96	0	0		0.000	
Sodium meta-silicate	Na ₂ SiO ₃ ·9H ₂ O	284.2	0.567	0.001995074	0.323	0.244	0.004
Silver Nitrate	AgNO ₃	169.87	0	0		0.000	
Strontium Nitrate	Sr(NO ₃) ₂	211.63	0	0		0.000	
Zinc Nitrate	Zn(NO ₃) ₂ ·6H ₂ O	297.47	0	0	0.000	0.000	
Zirconyl Nitrate		249.23	0	0		0.000	
Sodium Acetate	NaCH ₃ COO·3H ₂ O	136.08	2.42	0.017783657	0.961	1.459	0.018
Sodium Bromide	NaBr	102.89	0	0		0.000	0.000
Sodium Carbonate	Na ₂ CO ₃	105.99	82.914	0.782281347		82.914	1.565
Sodium Chloride	NaCl	58.44	0	0		0.000	0.000
Sodium Fluoride	NaF	41.99	3.028	0.072112408		3.028	0.072
Sodium Formate	HCOONa	68.01	10.983	0.161490957		10.983	0.161
Glycolic Acid	HOCH ₂ COOH, 70 wt%	76.05	19.632	0.18070217	5.890	13.742	
Citric Acid	C ₆ H ₈ O ₇ ·H ₂ O	210.14	7.56	0.035976016	0.648	6.912	
Sodium Nitrite	NaNO ₂	69	74.151	1.074652174		74.151	1.075
Sodium Oxalate	Na ₂ C ₂ O ₄	134	7.041	0.052544776		7.041	0.105
Sodium Phosphate	Na ₃ PO ₄ ·12H ₂ O	380.12	0	0	0.000	0.000	0.000
Sodium Sulfate	Na ₂ SO ₄	142.04	38.89	0.273796114		38.890	0.548
Sodium Nitrate	NaNO ₃	84.99	27.359	0.32190846		27.359	0.322
Sodium Hydroxide	NaOH	40	19.083	0.477075		19.083	0.477
Totals			311.794		8.001	303.793	4.388
Total Solution Mass		1206.6	grams				
Density		1.207	g/mL				
wt % solids		25.18	%				
Na Concentration		4.388	Molar				